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FINAL

CSF Negative Exposure Assessment Report - June

Libby, Montana Asbestos Project

Sample Processing

September 3, 2003

Contract No. DTRS57-99-D-00017
Task Order No. 20

Prepared for:

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Acronyms

AHERA	Asbestos Hazard Emergency Response Act of 1986
ASTM	American Society for Testing and Materials
CDM	CDM Federal Programs Corporation
cm^2	centimeter squared
CSF	close support facility
DQO	data quality objective
EPA	U.S. Environmental Protection Agency
EXC	excursion
f/cc	fibers per cubic centimeter
f/mm^2	fibers per square millimeter
H&S	health and safety
HEPA	high efficiency particulate air
Hygeia	Hygeia Laboratories Inc.
LA	Libby amphibole
L/min	liters per minute
NIOSH	National Institute for Occupational Safety and Health
OSHA	Occupational Safety and Health Administration
PCM	phase contrast microscopy
QC	quality control
s/cc	structures per cubic centimeter
s/cm^2	structures per square centimeter
SOP	standard operating procedure
TEM	transmission electron microscopy
TWA	time weighted average
Volpe	U.S. Department of Transportation Volpe Center
>	greater than
≥	greater than or equal to
≤	less than or equal to
µm	micron

Section 1
Introduction

In accordance with the soil preparation plan (CDM Federal Programs Corporation [CDM] 2003), a task-based negative exposure assessment was conducted between June 26 and June 30, 2003 to determine potential exposures at the CDM close support facility (CSF). The purpose of this report is to present the results of that assessment and the corrective actions taken.

1.1 Soil Sample Processing

CDM has been tasked by the U.S. Department of Transportation Volpe Center (Volpe) to prepare soil samples collected at the Libby site prior to their analysis. The preparation includes drying, sieving, splitting, and grinding. These procedures were developed to produce a sample with well-homogenized material of a standard particle size.

1.2 CDM Close Support Facility Location and Description

CDM prepares soil samples collected in Libby at its CSF located in Denver, Colorado. The CSF consists of approximately 3,000 square feet of space, which includes an office, drying room, wet chemistry room, storage/receiving room, equipment storage room, and the main laboratory.

Both ventilation hoods and the drying oven are vented to a high efficiency particulate air (HEPA) filter unit designed to remove 99.97 percent of particles 0.3 microns (μm) or greater. Figure 1-1 provides a floor plan of the CSF.

1.3 Close Support Facility Activities

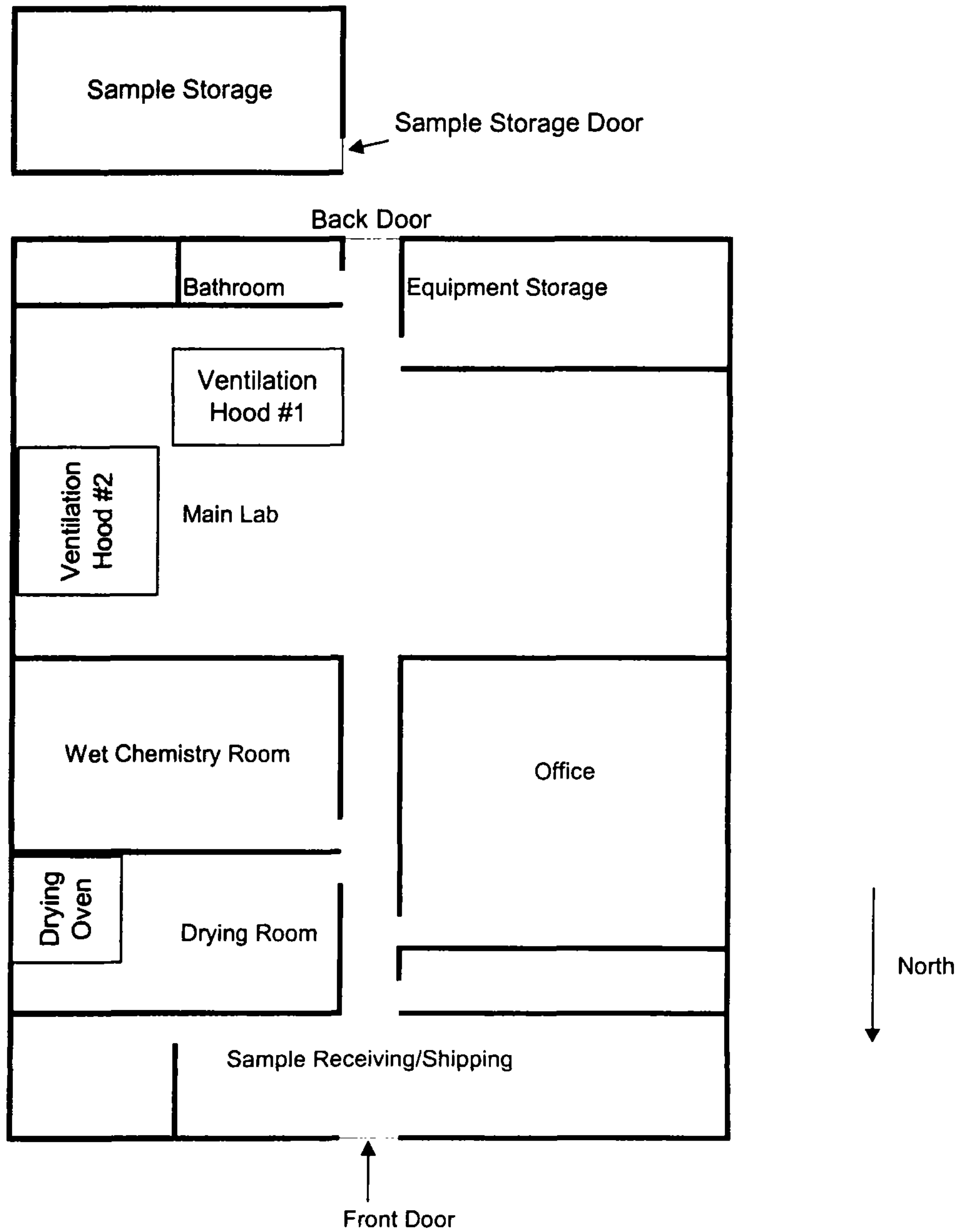
The following is a list of activities that are performed by CDM personnel at the CSF:

- Sample receipt and check-in
- Sample storage
- Sample drying
- Archive sample splitting
- Sample sieving
- Fine sample grinding
- Fine sample splitting and archiving

- Sample packaging and shipping
- CSF measurements
- Documentation
- Equipment decontamination

All sample preparation procedures are described in the Close Support Facility Soil Preparation Plan (CDM 2003).

Figure 1-1 CSF Floor Plan



Section 2
Negative Exposure Assessment

In accordance with the soil preparation plan (CDM 2003), a task-based negative exposure assessment was conducted to assess potential exposures and to document facility cleanliness at the CSF. The assessment consisted of ambient air samples, personal air samples, and microvacuum dust samples. During the negative exposure assessment, CSF personnel were in modified level-D personal protective equipment. CDM's CSF personnel performed the sampling. Hygeia Laboratories Inc. (Hygeia) performed the analysis.

2.1 Air Samples

2.1.1 Ambient Air Samples

Four ambient air samples were collected on June 30, 2003. Sampling locations included the office, sample receiving, main laboratory, and sample storage (Figure 2-1). Samples were collected using high-volume pumps at flow rates ranging between 8.51 and 8.55 liters per minute (L/min). Pumps were calibrated in accordance with section 7.2.3 of U.S. Environmental Protection Agency (EPA) standard operating procedure (SOP) 2015 *Asbestos Sampling* (EPA 1994) (Appendix B). Once calibrated, pumps were placed either on stands or on tables, with cassettes positioned downward, and sampling was conducted in accordance with section 7.4.2 of EPA SOP 2015.

2.1.2 Personal Air Samples

Personal air samples were collected by task (i.e., activity) to allow for differentiation among various exposure scenarios within the CSF. The two tasks sampled were sample receiving/sample coordinator and sample preparation. The sample receiving/sample coordinator position is responsible for checking in samples from the field, sample shipping, and general office activities. The sample preparation position is responsible for sieving, grinding, and splitting samples. Each task was sampled for 3 consecutive processing days. During each day, a time-weighted average (TWA) and a 30-minute excursion sample were collected for each task. TWA samples were collected using personal air sampling pumps at flow rates ranging between 2.03 and 2.1 L/min. Thirty minute excursion samples were collected using personal air sampling pumps at flow rates ranging between 2.03 and 2.13 L/min.

2.1.3 Air Sample Analysis

Ambient air samples were analyzed by: Appendix A to Subpart E of Part 763 (AHERA 2002).

Personal air samples were analyzed by:

1. National Institute for Occupational Safety and Health (NIOSH) Method 7400 (NIOSH 1994)

2. Appendix A to Subpart E of Part 763 (AHERA 2002)

2.2 Dust Samples

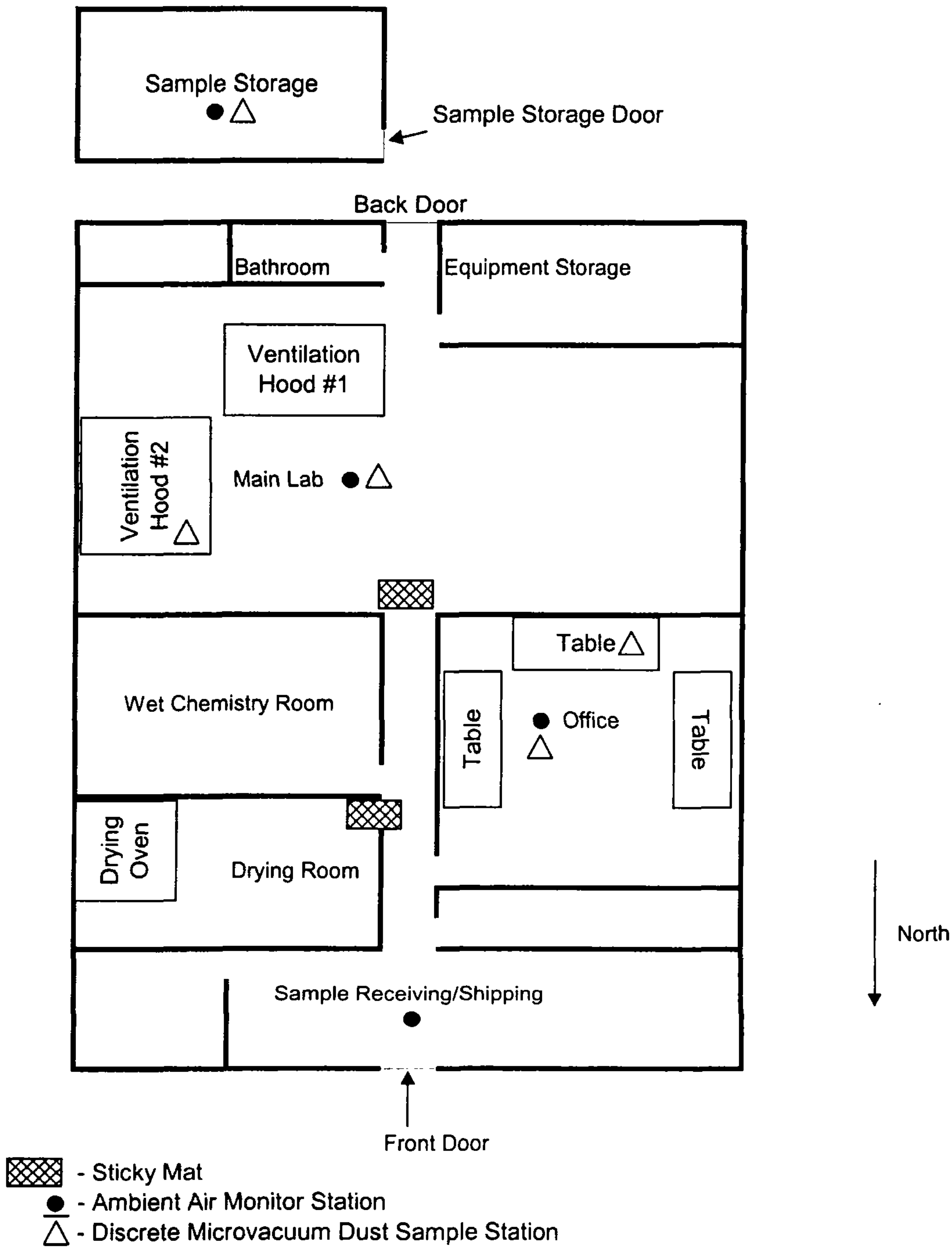
2.2.1 Microvacuum Dust Samples

Six microvacuum dust samples were collected on May 30, 2003. Samples were collected from tables in the office, the office floor, inside ventilation hood #2, the main laboratory floor, top of boxes in sample storage, and the sample storage floor (Figure 2-1). Samples were collected using personal air sampling pumps at a flow rate of 2.1 L/min. A composite sample consisting of three independent 100 square centimeter (cm^2) grids were sampled for 2 minutes each for a total of 300 cm^2 and 6 minutes per sampling location (composite dust sample). All samples were collected in accordance with American Society for Testing and Materials (ASTM) Standard D 5755-95 (ASTM 1995.

2.2.2 Dust Sample Analysis

All dust samples were analyzed using transmission electron microscopy (TEM), which stipulates the AHERA counting protocol (ASTM D5755-95).

Figure 2-1 Negative Exposure Assessment Sampling Locations



Section 3
Quality Assurance

The quality assurance for this negative exposure assessment will be discussed in the following four sections: deviations from the sampling and analysis plan, usability of the data, achievement of data quality objectives (DQOs), and summary of quality control (QC) activities.

3.1 Deviations from the Sampling and Analysis Plan

Deviation #1
The soil preparation plan calls for ambient air samples to be collected in close proximity to the following areas:

1) Sample receiving/shipping
2) Soil drying room
3) Sample preparation area in the main laboratory
4) Office

During the June negative exposure assessment, ambient samples were collected in close proximity to all of the aforementioned areas except the soil drying room. In addition, an ambient air sample was collected in the sample storage area.

No drying had occurred since the May negative exposure assessment and none was scheduled during the week of the June negative exposure assessment. Therefore, CDM decided to forgo sampling this area and focus on areas where operations were taking place. This included collecting an ambient air sample in the newly added sample storage area.

Deviation #2
The soil preparation plan calls for microvacuum dust samples to be collected from the following areas:

1) Inside the ventilation hood
2) Soil drying room
3) Sample preparation area in the main laboratory
4) Office

As mentioned above, the soil drying room was not used between the May and June negative exposure assessment and, therefore, no samples were collected in this area. Instead, microvacuum dust samples were collected from two locations in the sample storage area (floor and top of boxes).

Deviation #3
The soil preparation plan calls for all air samples to be analyzed by both PCM (NIOSH 7400) and TEM (AHERA). The ambient air samples were mistakenly only submitted for TEM. All ambient TEM results were non-detect. Because TEM is a more sensitive analytical method, CDM determined that resubmitting these samples for PCM analysis would not provide any additional information.

3.2 Usability of the Data

None of the negative exposure assessment data were either evaluated or validated. Therefore, it is assumed that the raw data are usable for their intended purpose, which is to assess potential exposures and to document facility cleanliness at the CSF.

3.3 Achievement of Data Quality Objectives

Currently no formal DQO's exist for the CSF monitoring program, however the objectives of the negative exposure assessment, to assess the potential exposures and document facility cleanliness at the CSF, were achieved.

3.4 Summary of Quality Control Activities

The only QC samples collected during the negative exposure assessment were field blanks. Two blanks each of personal air, ambient air, and microvaccuum dust samples were collected. One blank from each sample type was analyzed while the second blank was archived.

All QC activities for the negative exposure assessment were completed in accordance with the soil preparation plan.

Section 4
Results

A sample collection key is presented in Table 4-1. A summary of all PCM sample results is presented in Table 4-2, and a summary of the TEM sample results is presented in Tables 4-3 (air) and 4-4 (dust). Detailed bench sheets are included as Appendix A.

Table 4-1. Negative Exposure Assessment Sample Collection Key

	Index Id	Sample Type	Sample Date	Personnel/Area	Duration (min)	Volume (L)	Area (cm^2)
Personal Air Samples	CS-12501	8-hour TWA	6/26/03	Sample prep.	550	1141	n/a
	CS-12502	30-min EXC	6/26/03	Sample prep.	30	63.9	n/a
	CS-12503	8-hour TWA	6/26/03	Sample coord.	550	1141	n/a
	CS-12504	30-min EXC	6/26/03	Sample coord.	30	63.9	n/a
	CS-12505	8-hour TWA	6/27/03	Sample prep.	480	996.0	n/a
	CS-12506	30-min EXC	6/27/03	Sample prep.	30	63.0	n/a
	CS-12507	8-hour TWA	6/27/03	Sample coord.	480	1012.8	n/a
	CS-12508	30-min EXC	6/27/03	Sample coord.	30	60.8	n/a
	CS-12509	8-hour TWA	6/30/03	Sample prep.	480	972.	n/a
	CS-12510	30-min EXC	6/30/03	Sample prep.	30	63.0	n/a
	CS-12511	8-hour TWA	6/30/03	Sample coord.	480	988.8	n/a
	CS-12512	30-min EXC	6/30/03	Sample coord.	30	61.5	n/a
Ambient Air Samples	CS-12515	Ambient air	6/30/03	Office	480	4104.0	n/a
	CS-12516	Ambient air	6/30/03	Sample receiving	480	4084.8	n/a
	CS-12517	Ambient air	6/30/03	Main laboratory	480	4099.2	n/a
	CS-12518	Ambient air	6/30/03	Sample storage	480	4104.0	n/a
Dust Samples	CS-12521	Microvacuum	6/30/03	Office (floor)	6*	n/a	300
	CS-12522	Microvacuum	6/30/03	Main Lab. (inside hood #2)	6*	n/a	300
	CS-12523	Microvacuum	6/30/03	Main Laboratory (floor)	6*	n/a	300
	CS-12524	Microvacuum	6/30/03	Sample storage (floor)	6*	n/a	300
	CS-12525	Microvacuum	6/30/03	Sample storage (top of boxes)	6*	n/a	300
	CS-12526	Microvacuum	6/30/03	Office (top of tables)	6*	n/a	300

* Microvacuum samples were collected in three independent 100 cm^2 (2 min. each) areas for a total of 300 cm^2 (6 min. total).
cm^2 – square centimeter, EXC – excursion, L – liter, min = minute, n/a – not applicable, TWA – time weighted average

Table 4-2 Summary of the Negative Exposure Assessment PCM Results

	ID Number	Sample Type	Sample Date	Personnel/Area	TWA (f/cc)	% of TWA PEL*	30 Minute Excursion (f/cc)	% of 30 Minute Excursion PEL**
Personal Air Samples	CS-12501	8-hour TWA	6/26/03	Sample prep.	0.006	6.00%	n/a	n/a
	CS-12502	30-min EXC	6/26/03	Sample prep.	n/a	n/a	0.054	5.40%
	CS-12503	8-hour TWA	6/26/03	Sample coord.	0.003	3.00%	n/a	n/a
	CS-12504	30-min EXC	6/26/03	Sample coord.	n/a	n/a	0.042	4.20%
	CS-12505	8-hour TWA	6/27/03	Sample prep.	0.012	12.00%	n/a	n/a
	CS-12506	30-min EXC	6/27/03	Sample prep.	n/a	n/a	0.062	6.20%
	CS-12507	8-hour TWA	6/27/03	Sample coord.	0.003	3.00%	n/a	n/a
	CS-12508	30-min EXC	6/27/03	Sample coord.	n/a	n/a	0.048	4.80%
	CS-12509	8-hour TWA	6/30/03	Sample prep.	0.010	10.00%	n/a	n/a
	CS-12510	30-min EXC	6/30/03	Sample prep.	n/a	n/a	0.047	4.70%
	CS-12511	8-hour TWA	6/30/03	Sample coord.	0.008	8.00%	n/a	n/a
	CS-12512	30-min EXC	6/30/03	Sample coord.	n/a	n/a	0.048	4.48%

TWA = time weighted average   EXC = excursion sample   f/cc = fibers per cubic centimeter   n/a = not applicable   PEL = permissible exposure limit
* Assumes no respiratory protection (PEL of 0.1 f/cc)   ** 30 minute excursion PEL is 1.0 f/cc   Analytical Method: NIOSH 7400, Rev. 3, Issue 2, 8/94
Hygeia Report #: 22887030016

Table 4-3 Summary of the Negative Exposure Assessment TEM Results (Air)

	ID Number	Sample Type	Sample Date	Personnel/Area	FP TEM (s/cc)	Asbestos Mineral	EXC TEM (s/cc)	Asbestos Type
	CS-12501	8-hour TWA	6/26/03	Sample prep.	<0.0048	ND	n/a	n/a
	CS-12502	30-min EXC	6/26/03	Sample prep.	n/a	n/a	<0.060	ND
	CS-12503	8-hour TWA	6/26/03	Sample coord.	<0.0048	ND	n/a	n/a
	CS-12504	30-min EXC	6/26/03	Sample coord.	n/a	n/a	<0.060	ND
	CS-12505	8-hour TWA	6/27/03	Sample prep.	<0.0048	ND	n/a	n/a
	CS-12506	30-min EXC	6/27/03	Sample prep.	n/a	n/a	<0.061	ND
	CS-12507	8-hour TWA	6/27/03	Sample coord.	<0.0048	ND	n/a	n/a
	CS-12508	30-min EXC	6/27/03	Sample coord.	n/a	n/a	<0.063	ND
	CS-12509	8-hour TWA	6/30/03	Sample prep.	<0.0050	ND	n/a	n/a
	CS-12510	30-min EXC	6/30/03	Sample prep.	n/a	n/a	<0.061	ND
	CS-12511	8-hour TWA	6/30/03	Sample coord.	<0.0049	ND	n/a	n/a
	CS-12512	30-min EXC	6/30/03	Sample coord.	n/a	n/a	<0.063	ND
Ambient Air Samples	CS-12515	Ambient air	6/30/03	Office	<0.0023	ND	n/a	n/a
	CS-12516	Ambient air	6/30/03	Sample receiving	0.0047	C	n/a	n/a
	CS-12517	Ambient air	6/30/03	Main laboratory	<0.0023	ND	n/a	n/a
	CS-12518	Ambient air	6/30/03	Sample storage	0.007	C	n/a	n/a

ND = none detected *n/a = not applicable* *s/cc = structures per cubic centimeter* FP = *full period sample* EXC = *excursion sample*
TWA = time weighted average TEM = transmission electron microscopy C = chrysotile Analytical Method: 40 CFR, Part 763, Appendix A to Subpart E, Final Rule and Notice, October 30, 1987, for the Asbestos Hazard Emergency Response Act (AHERA) of 1986 using TEM with SAED and EDXA. This method was modified to allow project specific requirements (Mod. No. LB-000017). Hygeia Report #: 22887030017

Table 4-4 Summary of the Negative Exposure Assessment TEM Results (Dust)

ID Number	Sample Type	Sample Date	Personnel/Area	Area (cm^2)	TEM (s/cm^2)	Asbestos Type
CS-12521	Microvacuum	6/30/03	Office (floor)	300	<173	ND
CS-12522	Microvacuum	6/30/03	Main Laboratory (inside hood #2)	300	<173	ND
CS-12523	Microvacuum	6/30/03	Main Laboratory (floor)	300	<173	ND
CS-12524	Microvacuum	6/30/03	Sample storage (floor)	300	<173	ND
CS-12525	Microvacuum	6/30/03	Sample storage (top of boxes)	300	<173	ND
CS-12526	Microvacuum	6/30/03	Office (top of tables)	300	<173	ND

s/cm^2 – structures per square centimeter cm^2 – square centimeter ND = none detected TEM = transmission electron miscroscopy

Analytical Method: ISO 10312 method dated 1995-05-01 using TEM with SAED and EDXA. The method was modified to allow particulate loading up to 25% (Mod. No. LB-000016. Hygeia Report #: 22887030018

Section 5
Findings and Corrective Actions

5.1 Findings

Results from the PCM analysis and TEM analysis for the ambient air samples and microvacuum dust samples were received from Hygeia on July 3, 2003. Personal air sample TEM results were received from Hygeia July 22, 2003.

5.1.1 PCM

Personal air samples were analyzed by PCM. The evaluation criteria, corrective action, and results for each are presented below. All evaluation criteria and corrective actions outlined in this section are described in the soil preparation plan (CDM 2003).

Personal Sample Results

Evaluation criteria: Time weighted average personal air samples are considered acceptable if the PCM result is less than or equal to (≤) 50 percent of the Occupational Safety and Health Administration (OSHA) permissible exposure limit (0.1 fibers per cubic centimeter [f/cc]). That is, action will be taken for any PCM result for TWA personal air samples greater than (>) 0.05 f/cc. Excursion samples are considered acceptable by PCM if the result is ≤ 50 percent of the OSHA permissible exposure limit (1.0 f/cc). That is, action will be taken for any PCM result for excursion samples of > 0.5 f/cc.

Corrective action: If either of the evaluation criteria is not met, the CSF will be re-cleaned by wet wiping and HEPA vacuuming the affected area. Personal air samples will then be recollected.

Results: No personal air PCM results were above the evaluation criteria. Therefore, no corrective action was taken based on these results.

5.1.2 TEM

All three types of samples (i.e., ambient air, personal air, and microvacuum dust) were analyzed by TEM. The evaluation criteria, corrective action, and results for each are presented below. All evaluation criteria and corrective actions outlined in this section are described in the soil preparation plan (CDM 2003).

Ambient

Evaluation criteria: Ambient air samples are considered acceptable if one or fewer Libby amphibole (LA) structures are detected.

Corrective action: If the evaluation criterion is not met, the CSF will be re-cleaned by wet wiping and HEPA vacuuming the affected area. Ambient air samples will then be recollected.

Results: No ambient air TEM results were above the evaluation criteria. Therefore, no corrective action was taken based on these results.

Personal Sample Results

Evaluation criteria: The action level for TEM analysis is 0.10 LA structures per cubic centimeter (s/cc) for structures between 0.5 microns and 5 microns and 0.01 s/cc for structures > 5 microns.

Corrective action: If either of these criteria is not met, the laboratory will be wet wiped and HEPA vacuumed, and personal air samples recollected.

Results: No personal air TEM results were above the evaluation criteria. Therefore, no corrective action was taken based on these results.

Microvacuum Results

Evaluation criteria: Microvacuum dust samples are considered acceptable if the TEM result is ≤ 5000 LA structures per square centimeter (s/cm^2).

Corrective action: If dust sample results indicate concentrations greater than 5,000 s/cm^2, the area represented by the sample will be wet wiped, HEPA vacuumed, and re-sampled.

Results: No microvacuum dust TEM results were above the evaluation criteria. Therefore, no corrective action was taken based on these results.

5.2 Corrective Actions

No corrective actions were taken based on the June negative exposure assessment results.

5.3 Process Improvements

Two facility changes have occurred at the CSF since the last negative exposure assessment report. These are (1) the addition of a second ventilation hood in the main laboratory and (2) sample storage was moved to a detached building behind the CSF. The second ventilation hood was added to increase sample processing productivity. Sample storage was moved to the detached building due to lack of room in the main laboratory.

Several procedural changes were made at the CSF following the May negative exposure assessment. A list of the changes along with the reason for the change follows:

Sample Re-bagging

Every sample stored at the CSF was re-bagged under a hood (i.e., the existing sample bag put into a second bag) and re-filed in new storage boxes. All old storage boxes were HEPA vacuumed and discarded.

In addition, all future samples will be re-bagged under a hood (i.e., existing sample bag will be put into a second bag) immediately following drying.

Reason for Change

During normal drying operations, the seals on the sample bags are damaged, thus, preventing the bags from being re-sealed after drying. Past practices at the CSF were to roll (i.e., not re-seal or re-bag) sample bags following drying and then place them into storage boxes. The potential of release of material during sample handling existed under the former practices.

Sample Drying

All future drying operations will be conducted within a negative flow ventilation hood that is vented to a HEPA filter unit designed to remove 99.97 percent of particles 0.3 μm or greater.

Reason for Change

Although the current drying oven was vented to a HEPA filter unit, the operation did not take place under negative pressure. Therefore, there was a potential of release of aterial during loading and unloading of the oven.

Section 6
Conclusions

None of the results from the June negative exposure assessment exceeded any of the evaluation criteria set forth in the soil preparation plan. This indicates that the procedural changes implemented at the CSF following the May assessment have prevented any further release of LA. Based on these data, the fibrous aerosol monitoring contingency will not be implemented, and the July monitoring will only include one day of assessment.

Section 7
References

AHERA 2002. Asbestos Hazard Emergency Response Act 40 CFR (Protection of Environment) Chapter I (Environmental Protection Agency) Subchapter R (Toxic Substances Control Act) Part 763 (Asbestos) Subpart E (Asbestos Containing Materials in Schools) Appendix A (Interim Transmission Electron Microscopy Analytical Methods - Mandatory and Nonmandatory - and Mandatory Section to Determine Completion of Response Actions). Source is the Federal Register 2 FR 41846, October, 1987 Data current as of the Federal Register Dated May 20, 2002.

ASTM 1995. Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations. ASTM D-5755-95

CDM. 2003. Libby Asbestos Site, Operable Unit 4. Libby, Montana. Close Support Facility Soil Preparation Plan. April.

EPA. 1994. Asbestos Sampling. Standard Operating Procedure #2015, Revision #0.0.

NIOSH 1994. NIOSH 7400 – Asbestos and Other Fibers by Phase Contrast Microscopy (PCM). NIOSH Manual of Analytical Methods, Fourth Edition, Revision #3. Issue 2. August 15.

TARGET SHEET

EPA REGION VIII

SUPERFUND DOCUMENT MANAGEMENT SYSTEM

DOCUMENT NUMBER: 2009435

SITE NAME: LIBBY ASBESTOS

DOCUMENT DATE: 09/03/2003

DOCUMENT NOT SCANNED

Due to one of the following reasons:

- ☐ PHOTOGRAPHS
- ☐ 3-DIMENSIONAL
- ☐ OVERSIZED
- ☐ AUDIO/VISUAL
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- ☐ POOR LEGIBILITY
- ☐ OTHER
- ☐ NOT AVAILABLE
- ☑ TYPES OF DOCUMENTS NOT TO BE SCANNED
  (Data Packages, Data Validation, Sampling Data, CBI, Chain of Custody)

DOCUMENT DESCRIPTION:

APPENDIX A Sample Bench Sheets

Contact the Superfund Records Center to view available document.
(303) 312-6473

Appendix B
U.S. EPA Standard Operating Procedure
2015. Asbestos Sampling

1.0 SCOPE AND APPLICATION

Asbestos has been used in many commercial products including building materials such as flooring tiles and sheet goods, paints and coatings, insulation, and roofing asphalts. These products and others may be found at hazardous waste sites hanging on overhead pipes, contained in drums, abandoned in piles, or as part of a structure. Asbestos tailing piles from mining operations can also be a source of ambient asbestos fibers. Asbestos is a known carcinogen and requires air sampling to assess airborne exposure to human health. This Standard Operating Procedure (SOP) provides procedures for asbestos air sampling by drawing a known volume of air through a mixed cellulose ester (MCE) filter. The filter is then sent to a laboratory for analysis. The U.S. Environmental Protection Agency/Environmental Response Team (U.S. EPA/ERT) uses one of four analytical methods for determining asbestos in air. These include: U.S. EPA's Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air for Transmission Electron Microscopy (TEM)[1]; U.S. EPA's Modified Yamate Method for TEM[2]; National Institute for Occupational Safety and Health (NIOSH) Method 7402 (direct method only) for TEM; and NIOSH Method 7400 for Phase Contrast Microscopy (PCM)[3]. Each method has specific sampling and analytical requirements (i.e., sample volume and flow rate) for determining asbestos in air.

The U.S. EPA/ERT typically follows procedures outlined in the TEM methods for determining mineralogical types of asbestos in air and for distinguishing asbestos from non-asbestos minerals. The Phase Contrast Microscopy (PCM) method is used by U.S. EPA/ERT as a screening tool since it is less costly than TEM. PCM cannot distinguish asbestos from non-asbestos fibers, therefore the TEM method may be necessary to confirm analytical results. For example, if an action level for the presence of fibers has been set and PCM analysis indicates that the action level has been exceeded, then TEM analysis can be used to quantify and identify asbestos structures through examination of their morphology crystal structures (through electron diffraction), and elemental composition (through energy dispersive X-ray analysis). In this instance samples should be collected for both analyses in side by side sampling trains (some laboratories are able to perform PCM and TEM analysis from the same filter). The Superfund method is designed specifically to provide results suitable for supporting risk assessments at Superfund sites, it is applicable to a wide range of ambient air situations at hazardous waste sites. U.S. EPA's Modified Yamate Method for TEM is also used for ambient air sampling due to high volume requirements. The PCM and TEM NIOSH analytical methods require lower sample volumes and are typically used indoors; however, ERT will increase the volume requirement for outdoor application.

Other Regulations pertaining to asbestos have been promulgated by U.S. EPA and OSHA. U.S. EPA's National Emission Standards for Hazardous Air Pollutants (NESHAP) regulates asbestos-containing waste materials. NESHAP establishes management practices and standards for the handling of asbestos and emissions from waste disposal operations (40 CFR Part 61, Subparts A and M). U.S. EPA's 40 CFR 763 (July 1, 1987)[4] and its addendum 40 CFR 763 (October 30, 1987)[4] provide comprehensive rules for the asbestos abatement industry. State and local regulations on these issues vary and may be more stringent than federal requirements. The OSHA regulations in 29 CFR 1910.1001 and 29 CFR 1926.58 specify work practices and safety equipment such as respiratory protection and protective clothing when handling asbestos. The OSHA standard for an 8-hour, time-weighted average (TWA) is 0.2 fibers/cubic centimeters of air. This standard pertains to fibers with a length-to-width ratio of 3 to 1 with a fiber length >5 µm[5,6]. An action level of 0.1 fiber/cc (one-half the OSHA standard) is the level U.S. EPA has established in which employers must initiate such activities as air monitoring, employee training, and medical surveillance [5,6].

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Prior to sampling, the site should be characterized by identifying on-site as well as off-site sources of airborne asbestos. The array of sampling locations and the schedule for sample collection, is critical to the success of an investigation. Generally, sampling strategies to characterize a single point source are fairly straightforward, while multiple point sources and area sources increase the complexity of the sampling strategy. It is not within the scope of this SOP to provide a generic asbestos air sampling plan. Experience, objectives, and site characteristics will dictate the sampling strategy.

During a site investigation, sampling stations should be arranged to distinguish spatial trends in airborne asbestos concentrations. Sampling schedules should be fashioned to establish temporal trends. The sampling strategy typically requires that the concentration of asbestos at the source (worst case) or area of concern (downwind), crosswind, as well as background (upwind) contributions be quantified. See Table 1 (Appendix A) for U.S. EPA/ERT recommended sampling set up for ambient air. Indoor asbestos sampling requires a different type of strategy which is identified in Table 2 (Appendix A). It is important to establish background levels of contaminants in order to develop a reference point from which to evaluate the source data. Field blanks and lot blanks can be utilized to determine other sources.

Much information can be derived from each analytical method previously mentioned. Each analytical method has specific sampling requirements and produce results which may or may not be applicable to a specific sampling effort. The site sampling objectives should be carefully identified so as to select the most appropriate analytical method. Additionally, some preparation (i.e., lot blanks results) prior to site sampling may be required, these requirements are specified in the analytical methods.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 Sample Preservation

No preservation is required for asbestos samples.

3.2 Sample Handling, Container and Storage Procedures

1. Place a sample label on the cassette indicating a unique sampling number. Do not put sampling cassettes in shirt or coat pockets as the filter can pick up fibers. The original cassette box is used to hold the samples.

2. Wrap the cassette individually in a plastic sample bag. Each bag should be marked indicating sample identification number, total volume, and date.

3. The wrapped sampling cassettes should be placed upright in a rigid container so that the cassette cap is on top and cassette base is on bottom. Use enough packing material to prevent jostling or damage. Do not use vermiculite as packing material for samples. If possible, hand carry to lab.

4. Provide appropriate documentation with samples (i.e., chain of custody and requested analytical methodology).

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Flow rates exceeding 16 liters/minute (L/min) which could result in filter destruction due to (a) failure of its physical support under force from the increased pressure drop; (b) leakage of air around the filter mount so that the filter is bypassed, or (c) damage to the asbestos structures due to increased impact velocities.

4.1 U.S. EPA's Superfund Method

4.1.1 Direct-transfer TEM Specimen Preparation Methods

Direct-Transfer TEM specimen preparation methods have the following significant interferences:

- The achievable detection limit is restricted by the particulate density on the filter, which in turn is controlled by the sampled air volume and the total suspended particulate concentration in the atmosphere being sampled.
- The precision of the result is dependent on the uniformity of the deposit of asbestos structures on the sample collection filter.
- Air samples must be collected so that they have particulate and fiber loadings within narrow ranges. If too high a particulate loading occurs on the filter, it is not possible to prepare satisfactory TEM specimens by a direct-transfer method. If too high a fiber loading occurs on the filter, even if satisfactory TEM specimens can be prepared, accurate fiber counting will not be possible.

4.1.2 Indirect TEM Specimen Preparation Methods

Indirect TEM specimen preparation methods have the following interferences:

- The size distribution of asbestos structures is modified.
- There is increased opportunity for fiber loss or introduction of extraneous contamination.
- When sample collection filters are ashed, any fiber contamination in the filter medium is concentrated on the TEM specimen grid.

It can be argued that direct methods yield an under-estimate of the asbestos structure concentration because many of the asbestos fibers present are concealed by other particulate material with which they are associated. Conversely, indirect methods can be considered to yield an over-estimate because some types of complex asbestos structures disintegrate during the preparation, resulting in an increase in the numbers of structures counted.

4.2 U.S. EPA's Modified Yamate Method for TEM

High concentrations of background dust interfere with fiber identification.

4.3 NIOSH Method for TEM

Other amphibole particles that have aspect ratios greater than 3:1 and elemental compositions similar to the asbestos minerals may interfere in the TEM analysis. Some non-amphibole minerals may give electron diffraction patterns similar to amphiboles. High concentrations of background dust interfere with fiber identification.

4.4 NIOSH Method for PCM

PCM cannot distinguish asbestos from non-asbestos fibers; therefore, all particles meeting the counting criteria are counted as total asbestos fibers. Fiber less than 0.25 um in length will not be detected by this method. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

5.0 EQUIPMENT/MATERIALS

5.1 Sampling Pump

The constant flow or critical orifice controlled sampling pump should be capable of a flow-rate and pumping time sufficient to achieve the desired volume of air sampled.

The lower flow personal sampling pumps generally provide a flow rate of 20 cubic centimeters/minute (cc/min) to 4 L/min. These pumps are usually battery powered. High flow pumps are utilized when flow rates between 2 L/min to 20 L/min are required. High flow pumps are used for short sampling periods so as to obtain the desired sample volume. High flow pumps usually run on AC power and can be plugged into a nearby outlet. If an outlet is not available then a generator should be obtained. The generator should be positioned downwind from the sampling pump. Additional voltage may be required if more than one pump is plugged into the same generator. Several electrical extension cords may be required if sampling locations are remote.

The recommended volume for the Superfund method (Phase I) requires approximately 20 hours to collect. Such pumps typically draw 6 amps at full power so that 2 lead/acid batteries should provide sufficient power to collect a full sample. The use of line voltage, where available, eliminates the difficulties associated with transporting stored electrical energy.

A stand should be used to hold the filter cassette at the desired height for sampling and the filter cassette shall be isolated from the vibrations of the pump.

5.2 Filter Cassette

The cassettes are purchased with the required filters in position, or can be assembled in a laminar flow hood or clean area. When the filters are in position, a shrink cellulose band or adhesive tape should be applied to cassette joints to prevent air leakage.

5.2.1 TEM Cassette Requirements

Commercially available field monitors, comprising 25 mm diameter three-piece cassettes, with conductive extension cowls shall be used for sample collection. The cassette must be new and not previously used. The cassette shall be loaded with an MCE filter of pore size 0.45 µm, and supplied from a lot number which has been qualified as low background for asbestos determination. The cowls should be constructed of electrically conducting material to minimize electrostatic effects. The filter shall be backed by a 5 µm pore size MCE filter (Figure 1, Appendix B).

5.2.2 PCM Cassette Requirements

NIOSH Method 7400, PCM involves using a 0.8 to 1.2 µm mixed cellulose ester membrane, 25 mm diameter, 50 mm conductive cowl on cassette (Figure 2, Appendix B). Some labs are able to perform PCM and TEM analysis on the same filter; however, this should be discussed with the laboratory prior to sampling.

5.3 Other Equipment

- Inert tubing with glass cyclone and hose barb
- Whirlbags (plastic bags) for cassettes
- Tools - small screw drivers
- Container - to keep samples upright
- Generator or electrical outlet (may not be required)
- Extension cords (may not be required)
- Multiple plug outlet
- Sample labels
- Air data sheets
- Chain of Custody records

6.0 REAGENTS

Reagents are not required for the preservation of asbestos samples.

7.0 PROCEDURES

7.1 Air Volumes and Flow Rates

Sampling volumes are determined on the basis of how many fibers need to be collected for reliable measurements. Therefore, one must estimate how many airborne fibers may be in the sampling location.

Since the concentration of airborne aerosol contaminants will have some effect on the sample, the following is a suggested criteria to assist in selecting a flow rate based on real-time aerosol monitor (RAM) readings in milligrams/cubic meter (mg/m^3).

	Concentration	Flow Rate
• Low RAM readings:	<6.0 mg/m^3	11-15. L/min
• Medium RAM readings:	>6.0 mg/m^3	7.5 L/min
• High RAM readings:	>10. mg/m^3	2.5 L/min

In practice, pumps that are available for environmental sampling at remote locations operate under a maximum load of approximately 12 L/min.

7.1.1 U.S. EPA's Superfund Method

The Superfund Method incorporates an indirect preparation procedure to provide flexibility in the amount of deposit that be can be tolerated on the sample filter and to allow for the selective concentration of asbestos prior to analysis. To minimize contributions to background contamination from asbestos present in the plastic matrices of membrane filters while allowing for sufficient quantities of asbestos to be collected, this method also requires the collection of a larger volume of air per unit area of filter than has traditionally been collected for asbestos analysis. Due to the need to collect large volumes of air, higher sampling flow rates are recommended in this method than have generally been employed for asbestos sampling in the past. As an alternative, samples may be collected over longer time intervals. However, this restricts the flexibility required to allow samples to be collected while uniform meteorological conditions prevail.

The sampling rate and the period of sampling should be selected to yield as high a sampled volume as possible, which will minimize the influence of filter contamination. Wherever possible, a volume of 15 cubic meters (15,000 L) shall be sampled for those samples intended for analysis only by the indirect TEM preparation method (Phase 1 samples). For those samples to be prepared by both the indirect and the direct specimen preparation methods (Phase 2 samples), the volumes must be adjusted so as to provide a suitably-loaded filter for the direct TEM preparation method. One option is to collect filters at several loadings to bracket the estimated optimum loading for a particular site. Such filters can be screened in the laboratory so that only those filters closest to optimal loading are analyzed. It has been found that the volume cannot normally exceed 5 cubic meters (5000 L) in an urban or agricultural area, and 10 cubic meters (10,000 L) in a rural area for samples collected on a 25 mm filter and prepared by a direct-transfer technique.

An upper limit to the range of acceptable flow rates for this method is 15 L/min. At many locations, wind patterns exhibit strong diurnal variations. Therefore, intermittent sampling (sampling over a fixed time interval repeated over several days) may be necessary to accumulate 20 hours of sampling time over constant wind conditions. Other sampling objectives also may necessitate intermittent sampling. The objective is to design a sampling schedule so that samples are collected under uniform conditions throughout the sampling interval. This method provides for such options. Air volumes collected on Phase I samples are maximized (<16 L/min). Air volumes collected on Phase 2 samples are limited to provide optimum loading for filters to be prepared by a direct-transfer procedure.

7.1.2 U.S. EPA's Modified Yamate Method for TEM

U.S. EPA's TEM method requires a minimum volume of 560 L and a maximum volume of 3,800 L in order to obtain an analytical sensitivity of 0.005 structures/cc. The optimal volume for TEM is 1200 L to 1800 L. These volumes are determined using a 200 mesh EM grid opening with a 25-mm filter cassette. Changes in volume would be necessary if a 37-mm filter cassette is used since the effective area of a 25 mm (385 sq mm) and 37 mm (855 sq mm) differ.

7.1.3 NIOSH Method for TEM and PCM

The minimum recommended volume for TEM and PCM is 400 L at 0.1 fiber/cc. Sampling time is adjusted to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for eight hours (700 to 2800 L) is appropriate in non-dusty atmospheres containing 0.1 fiber/cc. Dusty atmospheres i.e., areas with high levels of asbestos, require smaller sample volumes (<400 L) to obtain countable samples.

In such cases, take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3,000 to 10,000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If > 50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration. Do not exceed 0.5 mg total dust loading on the filter.

7.2 Calibration Procedures

In order to determine if a sampling pump is measuring the flow rate or volume of air correctly, it is necessary to calibrate the instrument. Sampling pumps should be calibrated immediately before and after each use. Preliminary calibration should be conducted using a primary calibrator such as a soap bubble type calibrator, (e.g., a Buck Calibrator, Gilibrator, or equivalent primary calibrator) with a representative filter cassette installed between the pump and the calibrator. The representative sampling cassette can be reused for calibrating other pumps that will be used for asbestos sampling. The same cassette lot used for sampling should also be used for the calibration. A sticker should be affixed to the outside of the extension cowl marked "Calibration Cassette."

A rotameter can be used provided it has been recently precalibrated with a primary calibrator. Three separate constant flow calibration readings should be obtained both before sampling and after sampling. Should the flow rate change by more than 5% during the sampling period, the average of the pre- and post-calibration rates will be used to calculate the total sample volume. The sampling pump used shall provide a non-fluctuating air-flow through the filter, and shall maintain the initial volume flow-rate to within ± 10% throughout the sampling period. The mean value of these flow-rate measurements shall be used to calculate the total air volume sampled. A constant flow or critical orifice controlled pump meets these requirements. If at any time the measurement indicates that the flow-rate has decreased by more than 30%, the sampling shall be terminated. Flexible tubing is used to connect the filter cassette to the sampling pump. Sampling pumps can be calibrated prior to coming on-site so that time is saved when performing on-site calibration.

7.2.1 Calibrating a Personal Sampling Pump with an Electronic Calibrator

1. See Manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 3, Appendix B) using a sampling pump, electronic calibrator, and a representative filter cassette. The same lot sampling cassette used for sampling should also be used for calibrating.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 foot) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the electronic calibrator.

4. Turn the electronic calibrator and sampling pump on. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

5. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

6. Perform the calibration three times until the desired flow rate of ± 5% is attained.

7.2.2 Calibrating a Rotameter with an Electronic Calibrator

1. See manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 4, Appendix B) using a sampling pump, rotameter, and electronic calibrator.

3. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.

4. Turn the electronic calibrator and sampling pump on.

5. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

6. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

7. Record the electronic calibrator flow rate reading and the corresponding rotameter reading. Indicate these values on the rotameter (sticker). The rotameter should be able to work within the desired flow range. Readings can also be calibrated for 10 cm^3 increments for Low Flow rotameters, 500 cm^3 increments for medium flow rotameters and 1 liter increments for high flow rotameters.

8. Perform the calibration three times until the desired flow rate of ± 5% is attained. Once on site, a secondary calibrator, i.e., rotameter may be used to calibrate sampling pumps.

7.2.3 Calibrating a Personal Sampling Pump with a Rotameter

1. See manufacturer's manual for Rotameter's Operational Instructions.

2. Set up the calibration train as shown in (Figure 5, Appendix B) using a rotameter, sampling pump, and a representative sampling cassette.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 ft) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the rotameter.

4. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.

5. Turn the sampling pump on.

6. Turn the flow adjust screw (or knob) on the personal sampling pump until the float ball on the rotameter is lined up with the precalibrated flow rate value. A sticker on the rotameter should indicate this value.

7. A verification of calibration is generally performed on-site in the clean zone immediately prior to the sampling.

7.3. Meteorology

It is recommended that a meteorological station be established. If possible, sample after two to three days of dry weather and when the wind conditions are at 10 mph or greater. Record wind speed, wind direction, temperature, and pressure in a field logbook. Wind direction is particularly important when monitoring for asbestos downwind from a fixed source.

7.4 Ambient Sampling Procedures

7.4.1 Pre-site Sampling Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.

2. Obtain necessary sampling equipment and ensure it is in working order and fully charged (if necessary).

3. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety plan.

4. Once on-site the calibration is performed in the clean zone. The calibration procedures are listed in Section 7.2.

5. After calibrating the sampling pump, mobilize to the sampling location.

7.4.2 Site Sampling

1. To set up the sampling train, attach the air intake hose to the cassette base. Remove the cassette cap (Figure 6 and 7, Appendix B). The cassette should be positioned downward, perpendicular to the wind

2. If AC or DC electricity is required then turn it on. If used, the generator should be placed 10 ft. downwind from the sampling pump.

3. Record the following in a field logbook: date, time, location, sample identification number, pump number, flow rate, and cumulative time.

4. Turn the pump on. Should intermittent sampling be required, sampling filters must be covered between active periods of sampling. To cover the sample filter: turn the cassette to face upward, place the cassette cap on the cassette, remove the inlet plug from the cassette cap, attach a rotameter to the inlet opening of the cassette cap to measure the flow rate, turn off the sampling pump, place the inlet plug into the inlet opening on the cassette cap. To resume sampling: remove the inlet plug, turn on the sampling pump, attach a rotameter to measure the flow rate, remove the cassette cap, replace the inlet plug in the cassette cap and invert the cassette, face downward and perpendicular to the wind.

5. Check the pump at sampling midpoint if sampling is longer than 4 hours. The generators may need to be regased depending on tank size. If a filter darkens in appearance or if loose dust is seen in the filter, a second sample should be started.

6. At the end of the sampling period, orient the cassette up, turn the pump off.

7. Check the flow rate as shown in Section 7.2.3. When sampling open-faced, the sampling cap should be replaced before post calibrating. Use the same cassette used for sampling for post calibration (increase dust/fiber loading may have altered the flow rate.

8. Record the post flow rate.

9. Record the cumulative time or run.

10. Remove the tubing from the sampling cassette. Still holding the cassette upright, replace the inlet plug on the cassette cap and the outlet plug on the cassette base.

7.4.3. Post Site Sampling

1. Follow handling procedures in Section 3.2, steps 1-4.

2. Obtain an electronic or hard copy of meteorological data which occurred during the sampling event. Record weather: wind speed, ambient temperature, wind direction, and precipitation. Obtaining weather data several days prior to the sampling event can also be useful.

7.5 Indoor Sampling Procedures

PCM analysis is used for indoor air samples. When analysis shows total fiber count above the OSHA action level 0.1 f/cc then TEM (U.S. EPA's Modified Yamate Method) is used to identify asbestos from non-asbestos fibers.

Sampling pumps should be placed four to five feet above ground level away from obstructions that may influence air flow. The pump can be placed on a table or counter. Refer to Table 2 (Appendix A) for a summary of indoor sampling locations and rationale for selection.

Indoor sampling utilizes high flow rates to increased sample volumes (2000 L for PCM and 2800 to 4200 L for TEM) in order to obtain lower detection limits below the standard, (i.e., 0.01 f/cc or lower [PCM] and 0.005 structures/cc or lower [TEM]).

7.5.1 Aggressive Sampling Procedures

Sampling equipment at fixed locations may fail to detect the presence of asbestos fibers. Due to limited air movement, many fibers may settle out of the air onto the floor and other surfaces and may not be captured on the filter. In the past, an 8-hour sampling period was recommended to cover various air circulation conditions. A quicker and more effective way to capture asbestos fibers is to circulate the air artificially so that the fibers remain airborne during sampling. The results from this sampling option typifies worst case condition. This is referred to as aggressive air sampling for asbestos. Refer to Table 2 for sample station locations.

1. Before starting the sampling pumps, direct forced air (such as a 1-horsepower leaf blower or large fan) against walls, ceilings, floors, ledges, and other surfaces in the room to initially dislodge fibers from surfaces. This should take at least 5 minutes per 1000 sq. ft. of floor.

2. Place a 20-inch fan in the center of the room. (Use one fan per 10,000 cubic feet of room space.) Place the fan on slow speed and point it toward the ceiling.

3. Follow procedures in Section 7.4.1 and 7.4.2 (Turn off the pump and then the fan(s) when sampling is complete.).

4. Follow handling procedures in Section 3.2, steps 1-4.

8.0 CALCULATIONS

The sample volume is calculated from the average flow rate of the pump multiplied by the number of minutes the pump was running (volume = flow rate X time in minutes). The sample volume should be submitted to the laboratory and identified on the chain of custody for each sample (zero for lot, field and trip blanks).

The concentration result is calculated using the sample volume and the numbers of asbestos structures reported after the application of the cluster and matrix counting criteria.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

Follow all QA/QC requirements from the laboratories as well as the analytical methods.

9.1 TEM Requirements

1. Examine lot blanks to determine the background asbestos structure concentration.

2. Examine field blanks to determine whether there is contamination by extraneous asbestos structures during specimen preparation.

3. Examine of laboratory blanks to determine if contamination is being introduced during critical phases of the laboratory program.

4. To determine if the laboratory can satisfactorily analyze samples of known asbestos structure concentrations, reference filters shall be examined. Reference filters should be maintained as part of the laboratory's Quality Assurance program.

5. To minimize subjective effects, some specimens should be recounted by a different microscopist.

6. Asbestos laboratories shall be accredited by the National Voluntary Laboratory Accreditation Program.

7. At this time, performance evaluation samples for asbestos in air are not available for Removal Program Activities.

9.2 PCM Requirements

1. Examine reference slides of known concentration to determine the analyst's ability to satisfactorily count fibers. Reference slides should be maintained as part of the laboratory's quality assurance program.

2. Examine field blanks to determine if there is contamination by extraneous structures during sample handling.

3. Some samples should be relabeled then submitted for counting by the same analyst to determine possible bias by the analyst.

4. Participation in a proficiency testing program such as the AIHA-NIOSH proficiency analytical testing (PAT) program.

10.0 DATA VALIDATION

Results of quality control samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results accordingly with the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety procedures. More specifically, when entering an unknown situation involving asbestos, a powered air purifying respirator (PAPR) (full face-piece) is necessary in conjunction with HEPA filter cartridges. See applicable regulations for action level, PEL, TLV, etc. If previous sampling indicates asbestos concentrations are below personal health and safety levels, then Level D personal protection is adequate.

12.0 REFERENCES

(1) Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air, Part 1: Method, EPA/540/2-90/005a, May 1990, and Part 2: Technical Background Document, EPA/540/2-90/005b, May 1990.

(2) Methodology for the Measurement of Airborne Asbestos by Electron Microscopy, EPA's Report No. 68-02-3266, 1984, G. Yamate, S.C. Agarwal, and R. D. Gibbons.

(3) National Institute for Occupational Safety and Health. NIOSH Manual of Analytical Method. Third Edition. 1987.

(4) U.S. Environmental Protection Agency. Code of Federal Regulations 40 CFR 763. July 1, 1987. Code of Federal Regulations 40 CFR 763 Addendum. October 30, 1987.

(5) U.S. Environmental Protection Agency. Asbestos-Containing Materials in Schools; Final Rule and Notice. 52 FR 41826.

(6) Occupational Safety and Health Administration. Code of Federal Regulations 29 CFR 1910.1001. Washington, D.C. 1987.

APPENDIX A

Tables

TABLE 1. SAMPLE STATIONS FOR OUTDOOR SAMPLING		
Sample Station Location	Sample Numbers	Rationale
Upwind/Background [1]	Collect a minimum of two simultaneous upwind/background samples 30 ° apart from the prevailing windlines.	Establishes background fiber levels.
Downwind	Deploy a minimum of 3 sampling stations in a 180 degree arc downwind from the source.	Indicates if asbestos is leaving the site.
Site Representative and/or Worst Case	Obtain one site representative sample which shows average condition on-site or obtain worst case sample (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

[1] More than one background station may be required if the asbestos originates from different sources.

TABLE 2

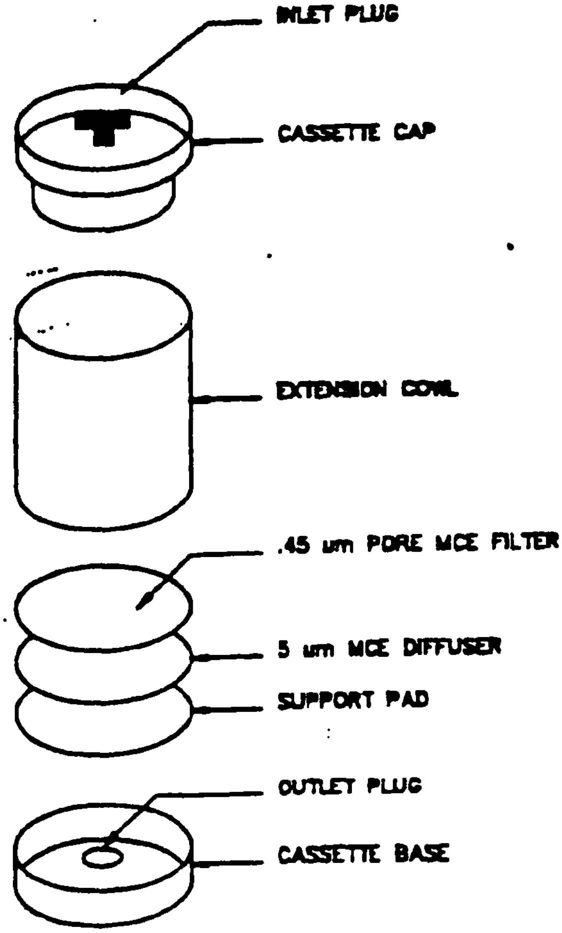
SAMPLE STATIONS FOR INDOOR SAMPLING

Sample Station Location	Sample Numbers	Rationale
Indoor Sampling	If a work site is a single room, disperse 5 samplers throughout the room. If the work site contains up to 5 rooms, place at least one sampler in each room. If the work site contains more than 5 rooms, select a representative sample of the rooms.	Establishes representative samples from a homogeneous area.
Upwind/Background	If outside sources are suspected, deploy a minimum of two simultaneous upwind/background samples 30 ° apart from the prevailing windlines.	Establish whether indoor asbestos concentrations are coming from an outside source.
Worst Case	Obtain one worst case sample, i.e., aggressive sampling (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

APPENDIX B

Figures

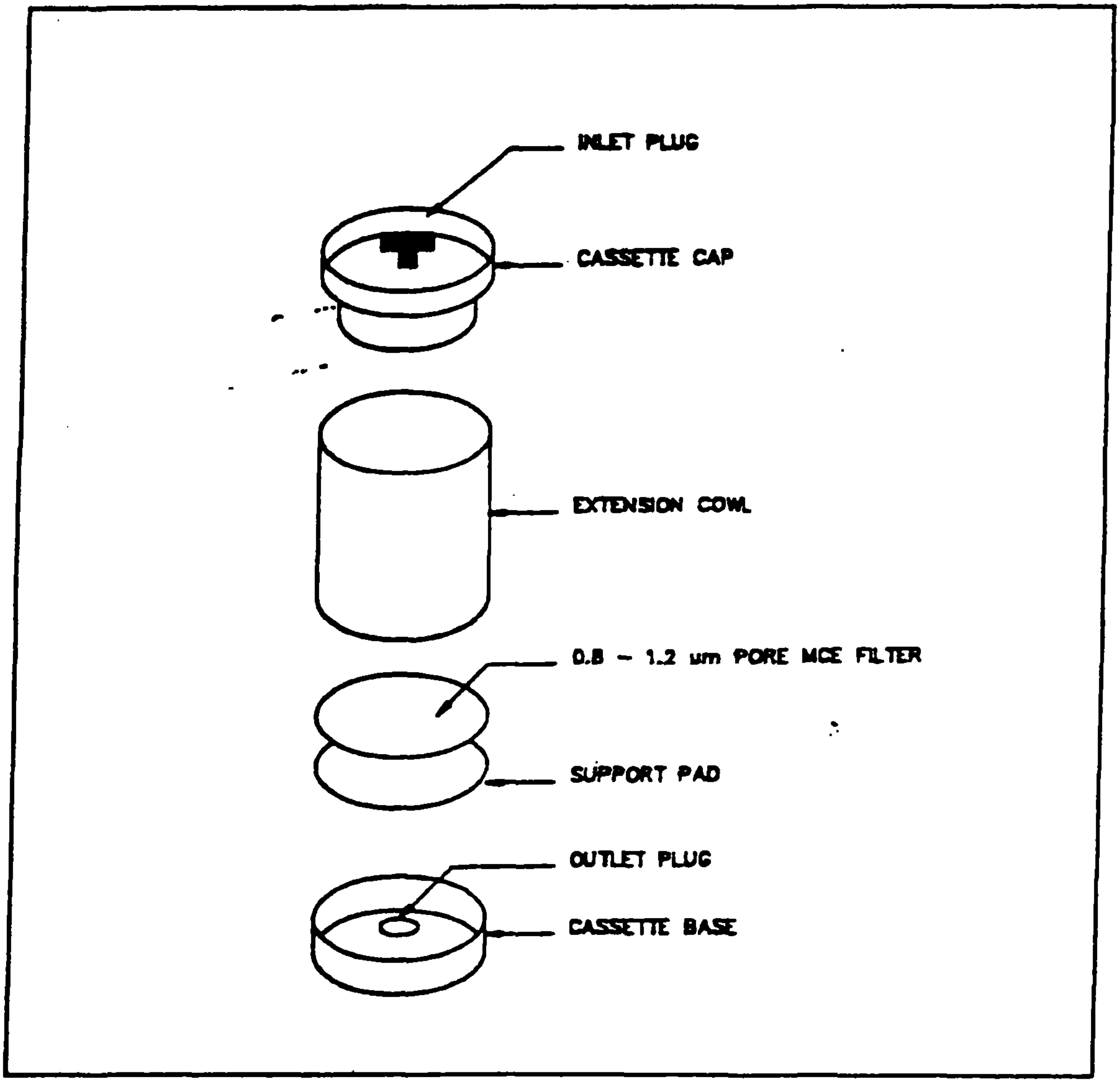
FIGURE 1. Transmission Electron Microscopy Filter Cassette



APPENDIX B (Cont'd)

Figures

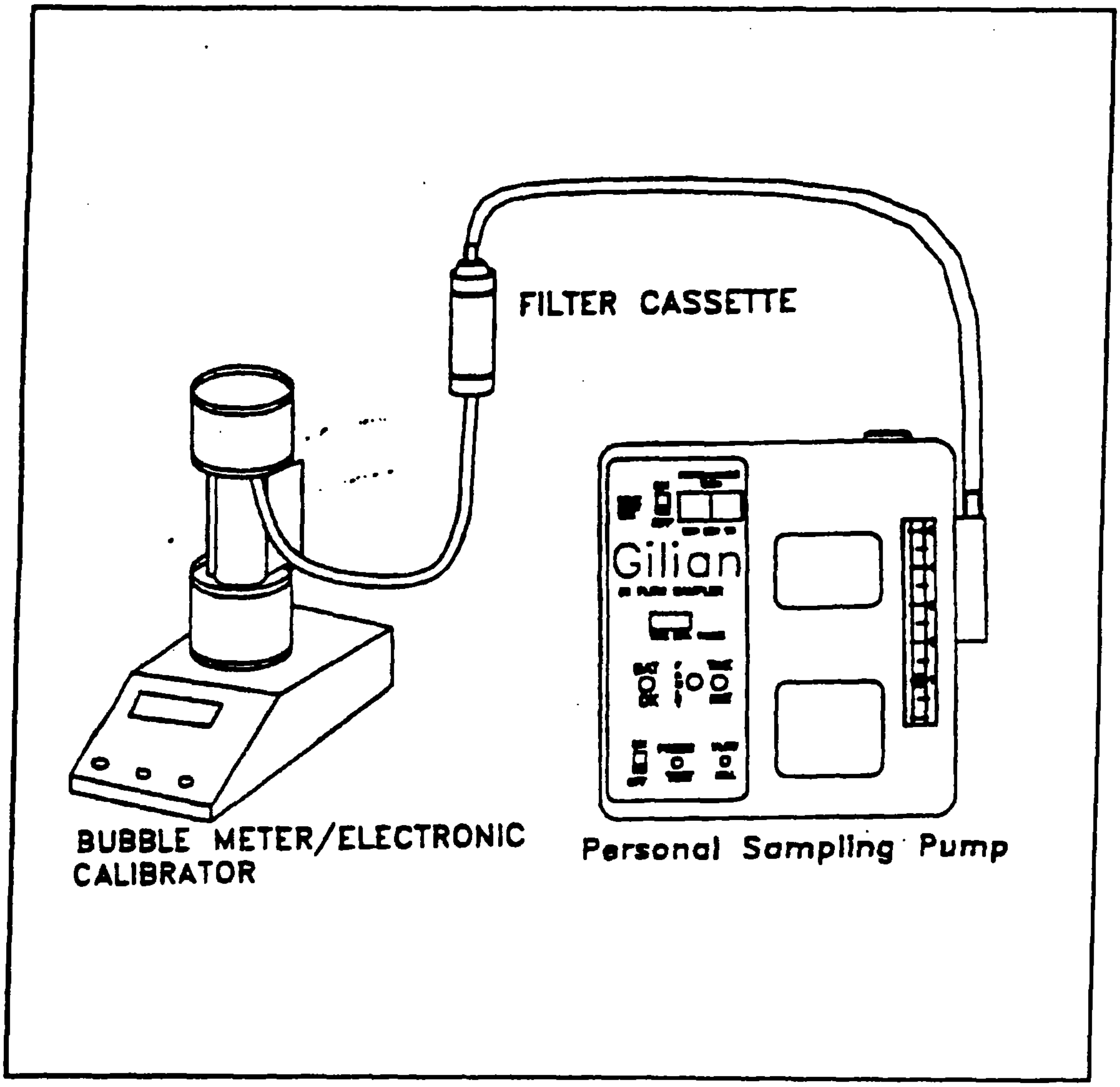
FIGURE 2. Phase Contrast Microscopy Filter Cassette



APPENDIX B (Cont'd)

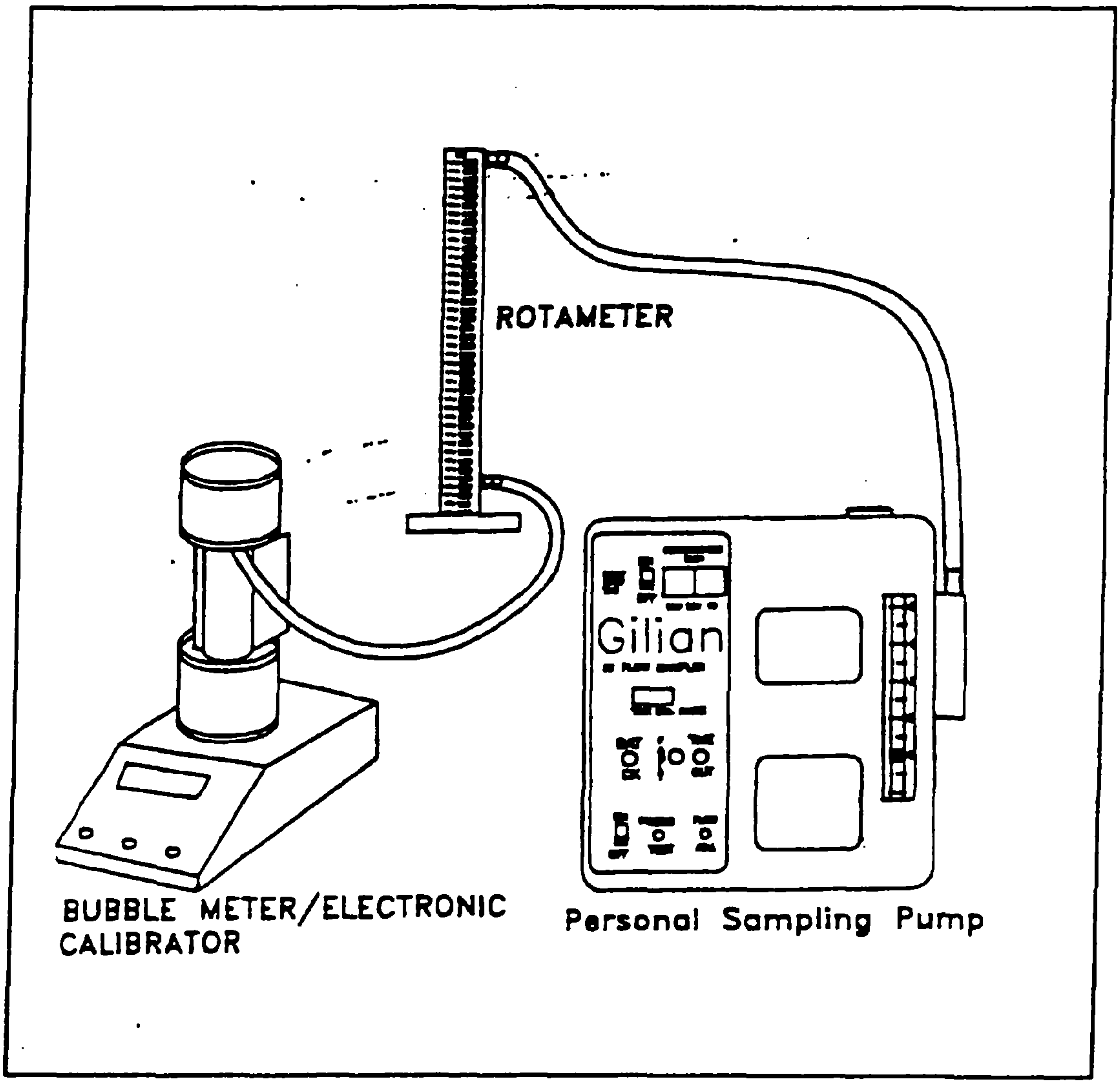
Figures

FIGURE 3. Calibrating a Personal Sampling Pump with a Bubble Meter



Figures

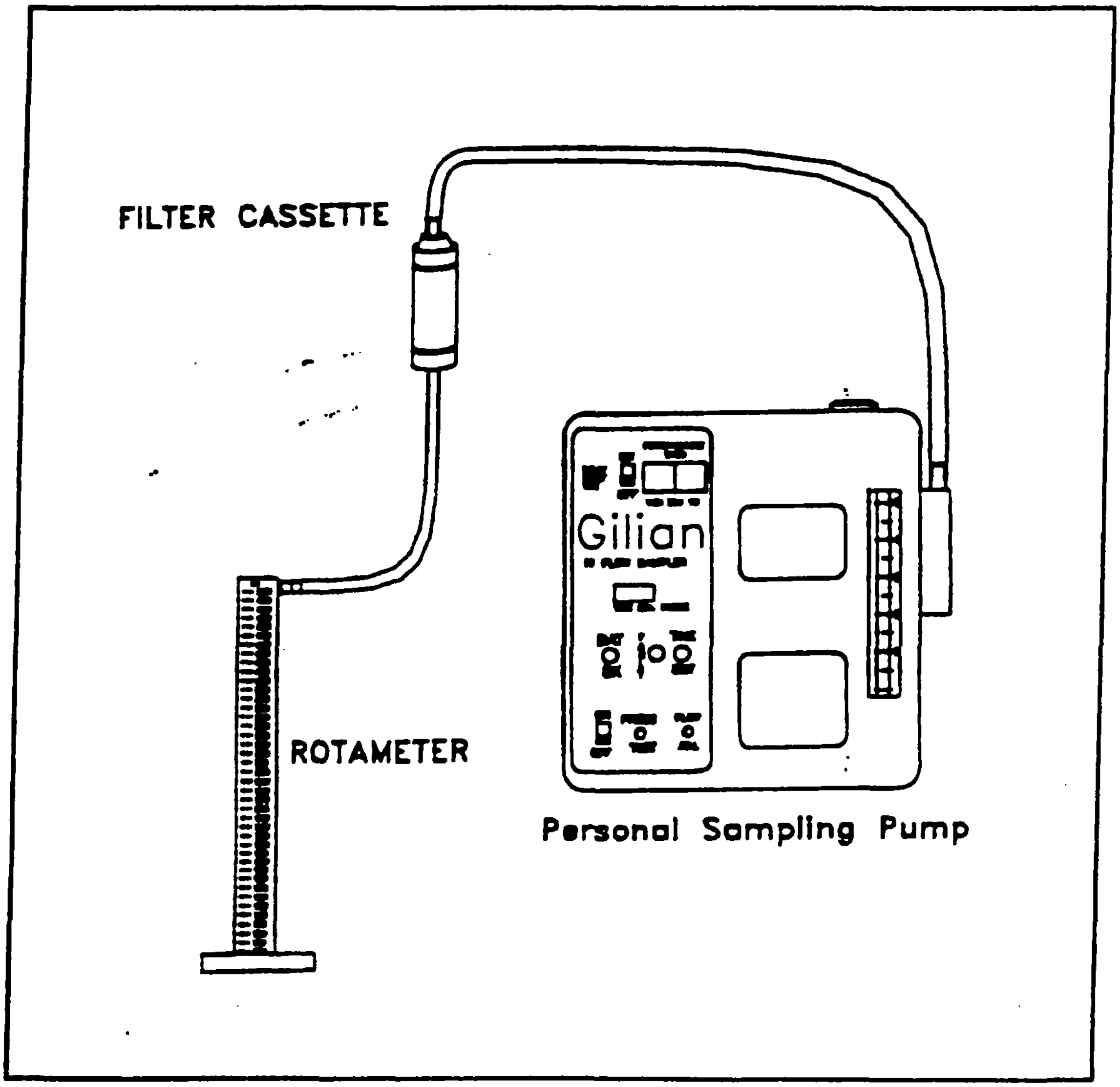
FIGURE 4. Calibrating a Rotameter with a Bubble Meter



APPENDIX B (Cont'd)

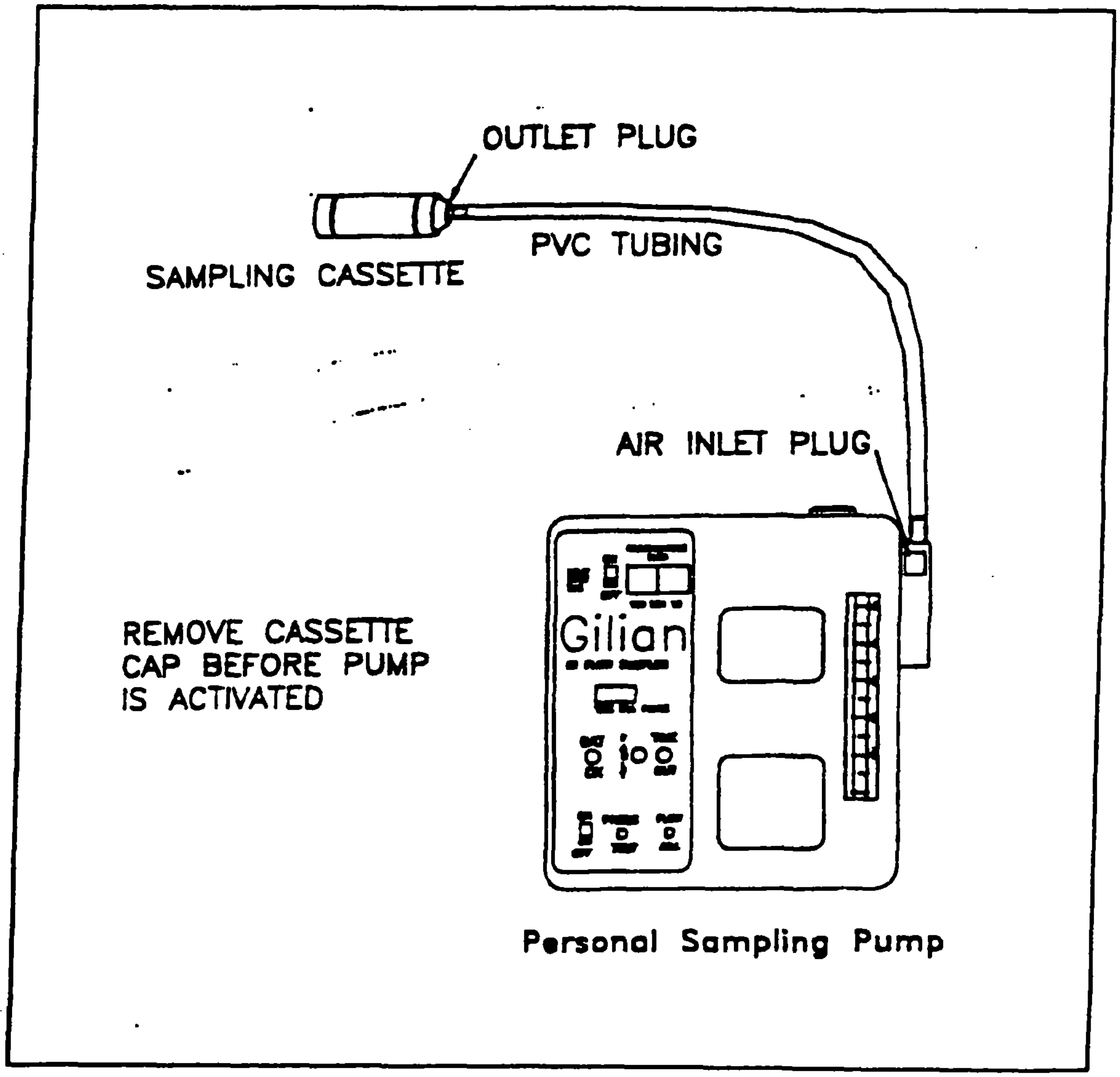
Figures

FIGURE 5. Calibrating a Sampling Pump with a Rotameter



Figures

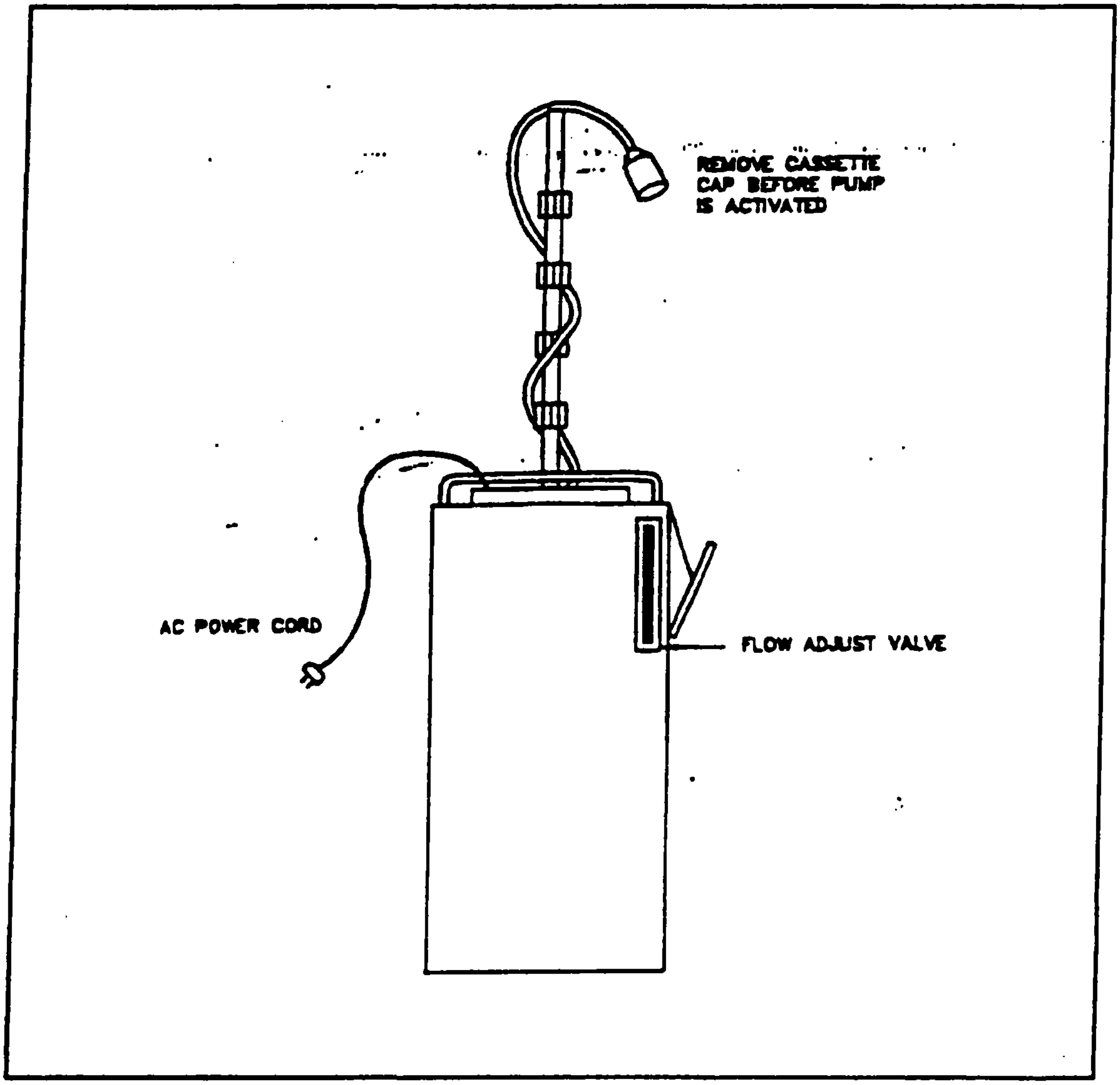
FIGURE 6. Personal Sampling Train for Asbestos



APPENDIX B (Cont'd)

Figures

FIGURE 7. High Flow Sampling Train for Asbestos



Appendix C
Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations. ASTM D-5755-95

Designation: D 5755 – 95

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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If not listed in the current combined index, will appear in the next edition.

Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations[1]

This standard is issued under the fixed designation D 5755; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure to (*a*) identify asbestos in dust and (*b*) provide an estimate of the concentration of asbestos in the sampled dust reported as the number of asbestos structures per unit area of sampled surface.

1.1.1 If an estimate of the asbestos mass is to be determined, the user is referred to Test Method D 5756.

1.2 This test method describes the equipment and procedures necessary for sampling, by a microvacuum technique, non-airborne dust for levels of asbestos structures. The non-airborne sample is collected inside a standard filter membrane cassette from the sampling of a surface area for dust which may contain asbestos.

1.2.1 This procedure uses a microvacuuming sampling technique. The collection efficiency of this technique is unknown and will vary among substrates. Properties influencing collection efficiency include surface texture, adhesiveness, electrostatic properties and other factors.

1.3 Asbestos identified by transmission electron microscopy (TEM) is based on morphology, selected area electron diffraction (SAED), and energy dispersive X-ray analysis (EDXA). Some information about structure size is also determined.

1.4 This test method is generally applicable for an estimate of the concentration of asbestos structures starting from approximately 1000 asbestos structures per square centimetre.

1.4.1 The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disperse aggregated asbestos into fundamental fibrils, fiber bundles, clusters, or matrices that can be more accurately quantified by transmission electron microscopy. However, as with all indirect sample preparation techniques, the asbestos observed for quantification may not represent the physical form of the asbestos as sampled. More specifically, the procedure described neither creates nor destroys asbestos, but it may alter the physical form of the mineral fibers.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1739 Test Method for the Collection and Measurement of Dustfall (Settleable Particulate Matter)[3]

D 3195 Practice for Rotameter Calibration[3]

D 3670 Guide for Determination of Precision and Bias of Methods of Committee D-22[3]

D 5756 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Concentration[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *asbestiform*—a special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral. For more information on asbestiform mineralogy, see Refs (1),[4] (2) and (3).

3.1.2 *asbestos*—a collective term that describes a group of naturally occurring, inorganic, highly fibrous, silicate dominated minerals, which are easily separated into long, thin, flexible fibers when crushed or processed.

DISCUSSION—Included in the definition are the asbestiform varieties of: serpentine (chrysotile); riebeckite (crocidolite); grunerite (grunerite asbestos); anthophyllite (anthophyllite asbestos); tremolite (tremolite asbestos); and actinolite (actinolite asbestos). The amphibole mineral compositions are defined according to nomenclature of the International Mineralogical Association (3).

Asbestos	Chemical Abstract Service No.[5]
Chrysotile	12001-29-5
Crocidolite	12001-28-4
Grunerite Asbestos	12172-73-5
Anthophyllite Asbestos	77536-67-5
Tremolite Asbestos	77536-68-6
Actinolite Asbestos	77536-66-4

3.1.3 *fibril*—a single fiber that cannot be separated into

[1] This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.07 on Sampling and Analysis of Asbestos.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.03.

[4] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[5] The non-asbestiform variations of the minerals indicated in 3.1.3 have different Chemical Abstract Service (CAS) numbers.

smaller components without losing its fibrous properties or appearance.

3.2 *Descriptions of Terms Specific to This Standard:*

3.2.1 *aspect ratio*—the ratio of the length of a fibrous particle to its average width.

3.2.2 *bundle*—a structure composed of three or more fibers in a parallel arrangement with the fibers closer than one fiber diameter to each other.

3.2.3 *cluster*—a structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings of fibers must have more than two points touching.

3.2.4 *debris*—materials that are of an amount and size (particles greater than 1 mm in diameter) that can be visually identified as to their source.

3.2.5 *dust*—any material composed of particles in a size range of ≤1 mm and large enough to settle by virtue of their weight from the ambient air (see definition for settleable particulate matter in Test Method D 1739).

3.2.6 *fiber*—a structure having a minimum length of 0.5 μm, an aspect ratio of 5:1 or greater, and substantially parallel sides (4).

3.2.7 *fibrous*—of a mineral composed of parallel, radiating, or interlaced aggregates of fibers, from which the fibers are sometimes separable. That is, the crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers, but has that distinct appearance. The term fibrous is used in a general mineralogical way to describe aggregates of grains that crystallize in a needle-like habit and appear to be composed of fibers. Fibrous has a much more general meaning than asbestos. While it is correct that all asbestos minerals are fibrous, not all minerals having fibrous habits are asbestos.

3.2.8 *indirect preparation*—a method in which a sample passes through one or more intermediate steps prior to final filtration.

3.2.9 *matrix*—a structure in which one or more fibers, or fiber bundles that are touching, are attached to, or partially concealed by a single particle or connected group of nonfibrous particles. The exposed fiber must meet the fiber definition (see 3.2.6).

3.2.10 *structures*—a term that is used to categorize all the types of asbestos particles which are recorded during the analysis (such as fibers, bundles, clusters, and matrices). Final results of the test are always expressed in asbestos structures per square centimetre.

4. Summary of Test Method

4.1 The sample is collected by vacuuming a known surface area with a standard 25 or 37 mm air sampling cassette using a plastic tube that is attached to the inlet orifice which acts as a nozzle. The sample is transferred from inside the cassette to an aqueous solution of known volume. Aliquots of the suspension are then filtered through a membrane. A section of the membrane is prepared and transferred to a TEM grid using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using SAED and EDXA at a magnification of 15 000 to 20 000X.

5. Significance and Use

5.1 This microvacuum sampling and indirect analysis method is used for the general testing of non-airborne dust samples for asbestos. It is used to assist in the evaluation of dust that may be found on surfaces in buildings such as ceiling tiles, shelving, electrical components, duct work, carpet, etc. This test method provides an index of the concentration of asbestos structures in the dust per unit area analyzed as derived from a quantitative TEM analysis.

5.1.1 This test method does not describe procedures or techniques required to evaluate the safety or habitability of buildings with asbestos-containing materials, or compliance with federal, state, or local regulations or statutes. It is the user's responsibility to make these determinations.

5.1.2 At present, a single direct relationship between asbestos-containing dust and potential human exposure does not exist. Accordingly, the user should consider these data in relationship to other available information in their evaluation.

5.2 This test method uses the definition, settleable particulate material, found in Test Method D 1739 as the definition of dust. This definition accepts all particles small enough to pass through a 1 mm (No. 18) screen. Thus, a single, large asbestos containing particle(s) (from the large end of the particle size distribution) dispersed during sample preparation may result in anomalously large asbestos concentration results in the TEM analyses of that sample. It is, therefore, recommended that multiple independent samples are secured from the same area, and a minimum of three samples analyzed by the entire procedure.

6. Interferences

6.1 The following minerals have properties (that is, chemical or crystalline structure) which are very similar to asbestos minerals and may interfere with the analysis by causing a false positive to be recorded during the test. Therefore, literature references for these materials must be maintained in the laboratory for comparison to asbestos minerals so that they are not misidentified as asbestos minerals.

6.1.1 *Antigorite.*

6.1.2 *Palygorskite (Attapulgite).*

6.1.3 *Halloysite.*

6.1.4 *Pyroxenes.*

6.1.5 *Sepiolite.*

6.1.6 *Vermiculite scrolls.*

6.1.7 *Fibrous talc.*

6.1.8 Hornblende and other amphiboles other than those listed in 3.1.2.

6.2 Collecting any dust particles greater than 1 mm in size in this test method may cause an interference and, therefore, must be avoided.

7. Materials and Equipment

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.[6]

7.2 *Transmission Electron Microscope (TEM)*, an 80 to 120 kV TEM, capable of performing electron diffraction, with a fluorescent screen inscribed with calibrated gradations, is required. The TEM must be equipped with energy dispersive X-ray spectroscopy (EDXA) and it must have a scanning transmission electron microscopy (STEM) attachment or be capable of producing a spot size of less than 250 nm in diameter in crossover.

7.3 *Energy Dispersive X-ray System (EDXA)*.

7.4 *High Vacuum Carbon Evaporator*, with rotating stage.

7.5 *High Efficiency Particulate Air (HEPA)*, filtered negative flow hood.

7.6 *Exhaust or Fume Hood*.

7.7 *Particle-free Water* (ASTM Type II, see Specification D 1193).

7.8 *Glass Beakers* (50 mL).

7.9 *Glass Sample Containers*, with wide mouth screw cap (200 mL) or equivalent sealable container (height of the glass sample container should be approximately 13 cm high by 6 cm wide).

7.10 *Waterproof Markers*.

7.11 *Forceps* (tweezers).

7.12 *Ultrasonic Bath*, table top model (100 W).

7.13 *Graduated Pipettes* (1, 5, 10 mL sizes), glass or plastic.

7.14 *Filter Funnel*, either 25 mm or 47 mm, glass or disposable. Filter funnel assemblies, either glass or disposable plastic, and using either a 25 mm or 47 mm diameter filter.

7.15 *Side Arm Filter Flask*, 1000 mL.

7.16 *Mixed Cellulose Ester (MCE) Membrane Filters*, 25 or 47 mm diameter, ≤0.22 µm and 5 µm pore size.

7.17 *Polycarbonate (PC) Filters*, 25 or 47 mm diameter, ≤0.2 µm pore size.

7.18 *Storage Containers*, for the 25 or 47 mm filters (for archiving).

7.19 *Glass Slides*, approximately 76 by 25 mm in size.

7.20 *Scalpel Blades*, No. 10, or equivalent.

7.21 *Cabinet-type Desiccator*, or low temperature drying oven.

7.22 *Chloroform*, reagent grade.

7.23 *Acetone*, reagent grade.

7.24 *Dimethylformamide (DMF)*.

7.25 *Glacial Acetic Acid*.

7.26 *1-methyl-2-pyrrolidone*.

7.27 *Plasma Asher*, low temperature.

7.28 *pH Paper*.

7.29 *Air Sampling Pump*, low volume personal-type, capable of achieving a flow rate of 1 to 5 L/min.

7.30 *Rotameter*.

7.31 *Air Sampling Cassettes*, 25 mm or 37 mm, containing 0.8 µm or smaller pore size MCE or PC filters.

7.32 *Cork Borer*, 7 mm.

7.33 *Non-Asbestos Mineral*, references as outlined in 6.1.

7.34 *Asbestos Standards*, as outlined in 3.1.2.

7.35 *Tygon*[7] *Tubing*, or equivalent.

7.36 *Small Vacuum Pump*, that can maintain a pressure of 92 kPa.

7.37 *Petri Dishes*, large glass, approximately 90 mm in diameter.

7.38 *Jaffe Washer*, stainless steel or aluminum mesh screen, 30 to 40 mesh, and approximately 75 mm by 50 mm in size.

7.39 *Copper TEM Finder Grids*, 200 mesh.

7.40 *Carbon Evaporator Rods*.

7.41 *Lens Tissue*.

7.42 *Ashless Filter Paper Filters*, 90 mm diameter.

7.43 *Gummed Paper Reinforcement Rings*.

7.44 *Wash Bottles*, plastic.

7.45 *Reagent Alcohol*, HPLC Grade (Fisher A995 or equivalent).

7.46 *Opening Mesh Screen*, plastic, 1.0 by 1.0 mm, (Spectra-Mesh #146410 or equivalent).

7.47 *Diffraction Grating Replica*.

8. Sampling Procedure for Microvacuum Technique

8.1 For sampling asbestos-containing dust in either indoor or outdoor environments, commercially available cassettes must be used. Air monitoring cassettes containing 25 mm or 37 mm diameter mixed cellulose ester (MCE) or polycarbonate (PC) filter membranes with a pore size less than or equal to 0.8 µm are required (7.31). The number of samples collected depends upon the specific circumstances of the study.

8.2 Maintain a log of all pertinent sampling information and sampling locations.

8.3 Sampling pumps and flow indicators shall be calibrated using a certified standard apparatus or assembly (see Practice D 3195 and 7.29).

8.4 Record all calibration information (5).

8.5 Perform a leak check of the sampling system at each sampling site by activating the pump (7.29) with the closed sampling cassette in line. Any air flow shows that a leak is present that must be eliminated before initiating the sampling operation.

8.6 Attach the sampling cassette to the sampling pump at the outlet side of the cassette with plastic tubing (7.35). The plastic tubing must be long enough in that the sample areas can be reached without interference from the sampling pump. Attach a clean, approximately 25.4 mm long piece of plastic tubing (6.35 mm internal diameter) directly to the inlet orifice. Use this piece of tubing as the sampling nozzle. Cut the sampling end of the tubing at a 45° angle as illustrated in Fig. 1. The exact design of the nozzle is not critical as long as some vacuum break is provided to avoid simply pushing the dust around on the surface with the nozzle rather than vacuuming it into the cassette. The internal diameter of the nozzle and flow rate of the pump may vary as long as the air velocity is 100 (±10) cm/s. This air velocity calculation is based on an internal sampling tube diameter of 6.35 mm at a flow rate of 2 L/min.

8.7 Measure and determine the sample area of interest. A

[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[7] Tygon is a registered trademark of the DuPont Co.

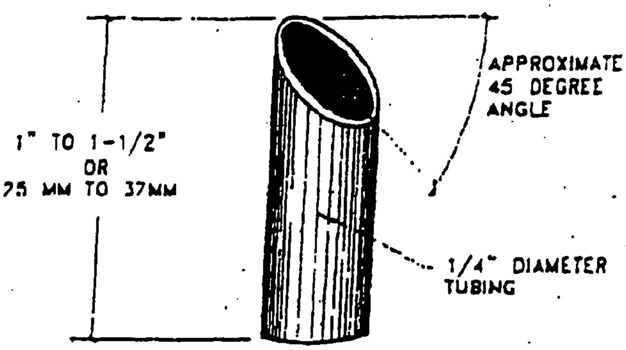

FIG. 1 Example of the Tubing Nozzle

sample area of 100 cm^2 is vacuumed until there is no visible dust or particulates matter remaining. Perform a minimum of two orthogonal passes on the surface within a minimum of 2 min of sampling time. Avoid scraping or abrading the surface being sampled. (Do not sample any debris or dust particles greater than 1 mm in diameter (see 4.2).) Smaller or larger areas can be sampled, if needed. For example, some surfaces of interest may have a smaller area than 100 cm^2. Less dusty surfaces may require vacuuming of larger areas. Unlike air samples, the overloading of the cassettes with dust will not be a problem. As defined in 3.2.5, only dust shall be collected for this analysis.

8.8 At the end of sample collection, invert the cassette so that the nozzle inlet faces up before shutting off the power to the pump. The nozzle is then sealed with a cassette end-plug and the cassette/nozzle taped or appropriately packaged to prevent separation of the nozzle and cassette assembly. A second option is the removal of the nozzle from the cassette, then plugging of the cassette and shipment of the nozzle (also plugged at both ends) sealed in a separate closeable plastic bag. A third option is placing the nozzle inside the cassette for shipment. The nozzle is always saved and rinsed because a significant percentage of the dust drawn from a lightly loaded surface may adhere to the inside walls of the tubing.

8.9 Check that all samples are clearly labeled, that all dust sampling information sheets are completed, and that all pertinent information has been enclosed, in accordance with laboratory quality control practices, before transfer of the samples to the laboratory. Include an unused cassette and nozzle as a field blank.

8.10 Wipe off the exterior surface of the cassettes with disposable wet towels (baby wipes) prior to packaging for shipment.

9. Sample Shipment

9.1 Ship dust samples to an analytical laboratory in a sealed container, but separate from any bulk or air samples. The cassettes must be tightly sealed and packed in a material free of fibers or dust to minimize the potential for contamination. Plastic "bubble pack" is probably the most appropriate material for this purpose.

10. Sample Preparation

10.1 Under a negative flow HEPA hood (7.5), carefully wet-wipe the exterior of the cassettes to remove any possible contamination before taking cassettes into a clean preparation area.

10.2 Perform sample preparation in a clean facility that has a separate work area from both the bulk and air sample preparation areas.

10.3 Initial specimen preparation shall take place in a clean HEPA filtered negative pressure hood to avoid any possible contamination of the laboratory or personnel, or both, by the potentially large number of asbestos structures in an asbestos-containing dust sample. Cleanliness of the preparation area hoods is measured by the cumulative process blank concentrations (see Section 11).

10.4 All sample preparation steps 10.4.1 through 10.4.6 shall take place in the dust preparation area inside a HEPA hood.

10.4.1 Remove the upper plug from the sample cassette and carefully introduce approximately 10 mL solution of a 50/50 mixture of particle-free water and reagent alcohol into the cassette using a plastic wash bottle (7.44). If the plugged nozzle was left attached to the cassette, then remove the plug and introduce the water/alcohol solution into the cassette through the tubing, and then remove the tubing, if it is visibly clean.

10.4.2 Replace the upper plug or the sample cap and lightly shake the dust suspension by hand for 3 s.

10.4.3 Remove the entire cap of the cassette and pour the suspension through a 1.0 by 1.0 mm opening screen (7.46) into a pre-cleaned 200 mL glass specimen bottle (7.9). All visible traces of the sample contained in the cassette shall be rinsed through the screen into the specimen bottle with a plastic wash bottle containing the 50/50 solution of particle-free water and alcohol. Repeat this procedure two additional times for a total of three washings. Next, rinse the nozzle two or three times through the screen into the specimen bottle with the 50/50 mixture of water and alcohol. Typically, the total amount of the 50/50 mixture used in the rinse is 50 to 75 mL. Discard the 1.0 by 1.0 mm screen and bring the volume of solution in the specimen bottle up to the 100 mL mark on the side of the bottle with particle-free water only.

10.4.4 Adjust the pH of the suspension to 3 to 4 using a 10.0 % solution of acetic acid. Use pH paper for testing. Filter the suspension within 24 h to avoid problems associated with bacterial and fungal growth.

10.4.5 Use either a disposable plastic filtration unit or a glass filtering unit (7.14) for filtration of aliquots of the suspension. The ability of an individual filtration unit to produce a uniform distribution may be tested by the filtration of a colored particulate suspension such as diluted India ink (suspension of carbon black).

10.4.5.1 If a disposable plastic filtration unit is used, then unwrap a new disposable plastic filter funnel unit (either 25 or 47 mm diameter) and remove the tape around the base of the funnel. Remove the funnel and discard the top filter supplied with the apparatus, retaining the coarse polypropylene support pad in place. Assemble the unit with the adapter and a properly sized neoprene stopper, and attach the funnel to the 1000 mL side-arm vacuum flask (7.15). Place a 5.0 µm pore size MCE (backing filter) on the support pad. Wet it with a few mL of particle-free water and place an MCE (7.16) or PC filter (≤0.22 µm pore size) (7.17) on top of the backing filter. Apply a vacuum (7.36), ensuring that the filters are centered and pulled flat without air bubbles. Any irregularities on the filter surface requires the discard of that filter. After the filter has been seated properly, replace the funnel and reseal it with the tape. Return the flask to atmospheric pressure.

10.4.5.2 If a glass filtration unit is used, place a 5 µm pore size MCE (backing filter) on the glass frit surface. Wet the filter with particle-free water, and place an MCE or PC filter (≤0.22 µm pore size) on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Replace the filters if any irregularities are seen on the filter surface. Before filtration of each set of sample aliquots, prepare a blank filter by filtration of 50 mL of particle-free water. If aliquots of the same sample are filtered in order of increasing concentration, the glass filtration unit need not be washed between filtration. After completion of the filtration, do not allow the filtration funnel assembly to dry because contamination is then more difficult to remove. Wash any residual suspension from the filtration assembly by holding it under a flow of water, then rub the surface with a clean paper towel soaked in a detergent solution. Repeat the cleaning operation, and then rinse two times in particle-free water.

10.4.6 With the flask at atmospheric pressure, add 20 mL of particle-free water into the funnel. Cover the filter funnel with its plastic cover if the disposable filtering unit is used.

10.4.7 Briefly hand shake (3 s) the capped bottle with the sample suspension, then place it in a tabletop ultrasonic bath (7.12) and sonicate for 3.0 min. Maintain the water level in the sonicator at the same height as the solution in sample bottle. The ultrasonic bath shall be calibrated as described in 20.5. The ultrasonic bath must be operated at equilibrium temperature. After sonicating, return the sample bottle to the work surface of the HEPA hood. Preparation steps 10.4.8 through 10.4.14 shall be carried out in this hood.

10.4.8 Shake the suspension lightly by hand for 3 s, then let it rest for 2.0 min to allow large particles to settle to the bottom of the bottle or float to the surface.

10.4.9 Estimate the amount of liquid to be withdrawn to produce an adequate filter preparation. Experience has shown that a light staining of the filter surface will yield a suitable preparation for analysis. Filter at least 1.0 mL, but no more than half the total volume. If after examination in the TEM, the smallest volume measured (1.0 mL) (7.13) yields an overloaded sample, then perform additional serial dilutions of the suspension. If it is estimated that less than 1.0 mL of solution has to be filtered because of the density of the suspension, perform a serial dilution.

10.4.9.1 If serial dilutions are required, repeat step 10.4.8 before the serial dilution portion is taken. Do not re-sonicate the original solution or any serial dilutions. The recommended procedure for a serial dilution is to mix 10 mL of the sample solution with 90 mL of particle-free water in a clean sample bottle to obtain a 1:10 serial dilution. Follow good laboratory practices when performing dilutions.

10.4.10 Insert a new disposable pipette halfway into the sample suspension and withdraw a portion. Avoid pipetting any of the large floating or settled particles. Uncover the filter funnel and dispense the mixture from the pipette into the water in the funnel.

10.4.11 Apply vacuum to the flask and draw the mixture through the filter.

10.4.12 Discard the pipette.

10.4.13 Disassemble the filtering unit and carefully remove the sample filter with fine tweezers (7.11). Place the completed sample filter particle side up, into a precleaned, labeled, disposable, plastic petri dish (7.48) or other similar container.

10.4.14 In order to ensure that an optimally-loaded filter is obtained, it is recommended that filters be prepared from several different aliquots of the dust suspension. For this series of filters, it is recommended that the volume of each aliquot of the original suspension be a factor of five higher than the previous one. If the filters are prepared in order of increasing aliquot volume, all of the filters for one sample can be prepared using one plastic disposable filtration unit, or without cleaning of glass filtration equipment between individual filtration. Before withdrawal of each aliquot from the sample, shake the suspension without additional sonification and allow to rest for 2 min.

10.4.15 There are many practical methods for drying MCE filters. The following are two examples that can be used: (1) dry MCE filters for at least 12 h (over desiccant) in an airtight cabinet-type desiccator (7.21); (2) to shorten the drying time (if desired), remove a plug of the damp filter and attach it to a glass slide (7.19) as described in 12.1.2 and 12.1.3. Place the slide with a filter plug or filter plugs (up to eight plugs can be attached to one slide) on a bed of desiccant, in the desiccator for 1 h.

10.4.16 PC filters do not require lengthy drying before preparation, but shall be placed in a desiccator for at least 30 min before preparation.

10.5 Prepare TEM specimens from small sections of each dried filter using the appropriate direct transfer preparation method.

11. Blanks

11.1 Prepare sample blanks that include both a process blank (50 mL of particle-free water) for each set of samples analyzed and one unused filter from each new box of sample filters (MCE or PC) used in the laboratory. If glass filtering units are used, prepare and analyze a process blank each time the filtering unit is cleaned. Blanks will be considered contaminated, if after analysis, they are shown to contain more than 53 asbestos structures per square millimetre. This generally corresponds to three or four asbestos structures found in ten grid openings. The source of the contamination must be found before any further analysis can be performed. Reject samples that were processed along with the contaminated blanks and prepare new samples after the source of the contamination is found.

11.2 Prepare field blanks which are included with sample sets in the same manner as the samples, to test for contamination during the sampling, shipping, handling, and preparation steps of the method.

12. TEM Specimen Preparation of Mixed Cellulose Ester (MCE) Filters

NOTE 1—Use of either the acetone or the diamethylformamide-acetic acid method is acceptable.

12.1 *Acetone Fusing Method:*

12.1.1 Remove a section (a plug) from any quadrant of the sample and blank filters. Sections can be removed from the filters using a 7 mm cork borer (7.32). The cork borer must be wet wiped after each time a section is removed.

12.1.2 Place the filter section (particle side up) on a clean microscope slide. Affix the filter section to the slide with a gummed page reinforcement (7.43), or other suitable means. Label the slide with a glass scribing tool or permanent marker (7.10).

12.1.3 Prepare a fusing dish from a glass petri dish (7.37) and a metal screen bridge (7.38) with a pad of five to six ashless paper filters (7.42) and place in the bottom of the petri dish (4). Place the screen bridge on top of the pad and saturate the filter pads with acetone. Place the slide on top of the bridge in the petri dish and cover the dish. Wait approximately 5 min for the sample filter to fuse and clear.

12.2 *Dimethylformamide-Acetic Acid Method:*

12.2.1 Place a drop of clearing solution that consists of 35 % dimethylformamide (DMF), 15 % glacial acetic acid, and 50 % Type II water (v/v) on a clean microscope slide. Gauge the amount used so that the clearing solution just saturates the filter section.

12.2.2 Carefully lay the filter segment, sample surface upward, on top of the solution. Bring the filter and solution together at an angle of about 20° to help exclude air bubbles. Remove any excess clearing solution. Place the slide in an oven or on a hot plate, in a fume hood, at 65 to 70°C for 10 min.

12.3 Plasma etching of the collapsed filter is required.

12.3.1 The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher (7.27). Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the exact conditions that must be used. Insufficient etching will result in a failure to expose embedded fibers, and too much etching may result in the loss of particles from the filter surface. To determine the optimum time for ashing, place an unused 25 mm diameter MCE filter in the center of a glass microscope slide. Position the slide approximately in the center of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 to 20 cm^3/min. Adjust the tuning of the system so that the intensity of the plasma is maximized. Determine the time required for complete oxidation of the filter. Adjust the system parameters to achieve complete oxidation of the filter in a period of approximately 15 min. For etching of collapsed filters, use these operating parameters for a period of 8 min. For additional information on calibration, see the *USEPA Asbestos-Containing Materials in Schools* (4) or *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* (6) documents.

12.3.2 Place the glass slide containing the collapsed filters into the low-temperature plasma asher, and etch the filter.

12.4 Carbon coating of the collapsed and etched filters is required.

12.4.1 Carbon coating must be performed with a high-vacuum coating unit (7.4), capable of less than 10^{-4} torr (13 MPa) pressure. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application and shall not be used. Carbon rods (7.40) used for evaporators shall be sharpened with a carbon rod sharpener to a neck of about 4 mm in length and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 100 to 120 mm from the surface of the microscope slide held in the rotating device.

12.4.2 Place the glass slide holding the filters on the rotation device, and evacuate the evaporator chamber to a vacuum of at least 13 MPa. Perform the evaporation in very short bursts, separated by 3 to 4 s to allow the electrodes to cool. An alternate method of evaporation is by using a slow continuous applied current. An experienced analyst can judge the thickness of the carbon film to be applied. Conduct tests on unused filters first. If the carbon film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen.

12.4.2.1 If the coating is too thick, it will lead to a TEM image that is lacking in contrast, and the ability to obtain electron diffraction patterns will be compromised. The carbon film shall be as thin as possible and still remain intact on most of the grid openings of the TEM specimen.

12.5 *Preparation of the Jaffe Washer*—The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used (7, 8). One such washer consists of a simple stainless steel bridge contained in a glass petri dish.

12.5.1 Place several pieces of lens tissue (7.41) on the stainless steel bridge. The pieces of lens tissue shall be large enough to completely drape over the bridge and into the solvent. In a fume hood, fill the petri dish with acetone (or DMF) until the height of the solvent is brought up to contact the underside of the metal bridge as illustrated in Fig. 2.

12.6 *Placing the Specimens into the Jaffe Washer:*

12.6.1 Place the TEM grids (7.39) shiny side up on a piece of lens tissue or filter paper so that individual grids can be easily picked up with tweezers.

12.6.2 Prepare three grids from each sample.

12.6.2.1 Using a curved scalpel blade (7.20), excise at least two square (3 mm by 3 mm) pieces of the carbon-coated MCE filter from the glass slide.

12.6.2.2 Place the square filter piece carbon-side up on top of a TEM specimen grid.

12.6.2.3 Place the whole assembly (filter/grid) on the saturated lens tissue in the Jaffe washer.

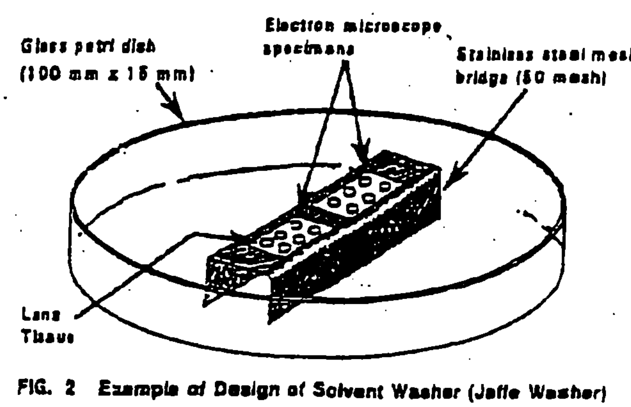

FIG. 2 Example of Design of Solvent Washer (Jaffe Washer)

12.6.2.4 Place the three TEM grid sample filter preparations on the same piece of lens tissue in the Jaffe washer.

12.6.2.5 Place the lid on the Jaffe washer and allow the system to stand for several hours.

12.7 Alternately, place the grids on a low level (petri dish filled to the ¼ mark) DMF Jaffe washer for 60 min. Add enough solution of equal parts DMF/acetone to fill the washer to the screen level. Remove the grids after 30 min if they have cleared, that is, all filter material has been removed from the carbon film, as determined by inspection in the TEM.

12.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean marked grid box.

13. TEM Specimen Preparation of Polycarbonate (PC) Filter

13.1 Cover the surface of a clean microscope slide with two strips of double-sided adhesive tape.

13.2 Cut a strip of filter paper slightly narrower than the width of the slide. Position the filter paper strip on the center of the length of the slide.

13.3 Using a clean, curved scalpel blade, cut a strip of the PC filter approximately 25 by 6 mm. Use a rocking motion of the scalpel blade to avoid tearing the filter. Place the PC strip particle side up on the slide perpendicular to the long axis of the slide. The ends of the PC strip must contact the double sided adhesive tape. Each slide can hold several PC strips. With a glass marker, label each PC strip with the individual sample number.

13.4 Carbon coat the PC filter strips as discussed in 12.4.2. PC filters do not require etching.

NOTE 2: Caution—Do not overheat the filter sections while carbon coating.

13.5 Prepare a Jaffe washer as described in 12.5, but fill the washer with chloroform or 1-methyl-2-pyrrolidone to the level of the screen.

13.6 Using a clean curved scalpel blade, excise three, 3-mm square filter pieces from each PC strip. Place the filter squares carbon side up on the shiny side of a TEM grid. Pick up the grid and filter section together and place them on the lens tissue in the Jaffe washer.

13.7 Place the lid on the Jaffe washer and rest the grids in place for at least 4 h. Best results are obtained with longer wicking times, up to 12 h.

13.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean, marked grid box.

14. Grid Opening Measurements

14.1 TEM grids must have a known grid opening area. Determine this area as follows:

14.2 Measure at least 20 grid openings in each of 20 random 75 to 100 μm (200-mesh) copper grids for a total of 400 grid openings for every 1000 grids used, by placing the 20 grids on a glass slide and examining them under the optical microscope. Use a calibrated graticule to measure the average length and width of the 20 openings from each of the individual grids. From the accumulated data, calculate the average grid opening area of the 400 openings.

14.3 Grid area measurements can also be made at the TEM at a calibrated screen magnification of between 15 000 and 20 000X. Typically measure one grid opening for each grid examined. Measure grid openings in both the x and y directions and calculate the area.

14.4 Pre-calibrated TEM grids are also acceptable for this test method.

15. TEM Method

15.1 Microscope settings: 80 to 120 kV, 15 000 to 20 000X screen magnification for analysis (7.2).

15.2 Analyze two grids for each sample. Analyze one-half of the sample area on one sample grid preparation and the remaining half on a second sample grid preparation.

15.3 *Determination of Specimen Suitability:*

15.3.1 Carefully load the TEM grid, carbon side facing up (in the TEM column) with the grid bars oriented parallel/perpendicular to the length of the specimen holder. Use a hand lens or loupe, if necessary. This procedure will line up the grid with the X and y translation directions of the microscope. Insert the specimen holder into the microscope.

15.3.2 Scan the entire grid at low magnification (250X to 1000X) to determine its suitability for high magnification analysis as specified in 15.3.3.

15.3.3 Grids are acceptable for analysis if the following conditions are met:

15.3.3.1 The fraction of grid openings covered by the replica section is at least 50 %.

15.3.3.2 Relative to that section of the grid covered by the carbon replica, the fraction of intact grid openings is greater than 50 %.

15.3.3.3 The fractional area of undissolved filter is less than 10 %.

15.3.3.4 The fraction of grid openings with overlapping or folded replica film is less than 50 %.

15.3.3.5 At least 20 grid openings, that have no overlapping or folded replica, are less than 5 % covered with holes and have less than 5 % opaque area due to incomplete filter dissolution.

15.4 *Determination of Grid Opening Suitability:*

15.4.1 If the grid meets acceptance criteria, choose a grid opening for analysis from various areas of the grid so that the entire grid is represented. Determine the suitability of each individual grid opening prior to the analysis.

15.4.2 The individual grid opening must have less than 5 % holes over its area.

15.4.3 Grid openings must be less than 25 % covered with particulate matter.

15.4.4 Grid openings must be uniformly loaded.

15.5 Observe and record the orientation of the grid at 80 to 150X, on a grid map record sheet along with the location of the grid openings that are examined for the analysis. If indexed grids are used, a grid map is not required, but the identifying coordinates of the grid square must be recorded.

16. Recording Data Rules

16.1 Record on the count sheet any continuous grouping of particles in which an asbestos fiber is detected. Classify asbestos structures as fibers, bundles, clusters, or matrices as defined in 5.2.

16.2 Use the criteria for fiber, bundle, cluster, and matrix identification, as described in the *USEPA Asbestos-Containing*

Materials in Schools document (4). Record, for each AHERA structure identified, the length and width measurements.

16.3 Record NSD (No Structures Detected) when no structures are detected in the grid opening.

16.4 Identify structures classified as chrysotile identified by either electron diffraction or X-ray analysis (7.3) and recorded on a count sheet. Verify at least one out of every ten chrysotile structures by X-ray analysis.

16.5 Structures classified as amphiboles by X-ray analysis and electron diffraction are recorded on the count sheet. For more information on identification, see Yamate, et al, (7) or Chatfield and Dillon (8).

16.6 Record a typical electron diffraction pattern for each type of asbestos observed for each group of samples (or a minimum of every five samples) analyzed. Record the micrograph number on the count sheet. Record at least one X-ray spectrum for each type of asbestos observed per sample. Attach the print-outs to the back of the count sheet. If the X-ray spectrum is stored, record the file and disk number on the count sheet.

16.7 *Counting Rules*:

16.7.1 At a screen magnification of between 15 000 and 20 000X evaluate the grids for the most concentrated sample loading; reject the sample if it is estimated to contain more than 50 asbestos structures per grid opening. Proceed to the next lower concentrated sample until a set of grids are obtained that have less than 30 asbestos structures per grid opening.

16.8 *Analytical Sensitivity*—An analytical sensitivity of approximately 1000 asbestos structures per square centimetre (calculated for the detection of a single asbestos structure) has been designed for this analysis. This sensitivity can be achieved by increasing the amount of liquid filtered, increasing the number of grid openings analyzed, or decreasing the size of the final filter. Occasionally, due to high particle loadings or high asbestos concentration, this analytical sensitivity cannot be practically achieved and stopping rules apply.

16.9 *Limit of Detection*—The limit of detection for this method is defined as, at a minimum, the counting of four asbestos structures during the TEM analysis. If less than four asbestos structures are counted during the analysis then the analytical result which will be reported will be less than the limit of detection and a "less than" sign (<) will appear before the number. All data shall be provided in the laboratory report.

16.10 *Stopping Rules*:

16.10.1 The analysis is stopped upon the completion of the grid square that achieves an analytical sensitivity of less than 1000 asbestos structures per square centimetre.

16.10.2 If an analytical sensitivity of 1000 asbestos structures per square centimetre cannot be achieved after analyzing ten grid openings then stop on grid opening No. 10 or the grid opening which contains the 100th asbestos structure, whichever comes first. A minimum of four grid squares shall be analyzed for each sample.

16.10.2.1 If the analysis is stopped because of the 100th structure rule, the entire grid square containing the 100th structure must be counted.

16.11 After analysis, remove the grids from the TEM, and replace them in the appropriate grid storage holder.

17. Sample Storage

17.1 The washed-out sample cassettes can be discarded after use.

17.2 Sample grids and unused filter sections (7.18) must be stored for a minimum of one year.

18. Reporting

18.1 Report the following information for each dust sample analyzed:

18.1.1 Concentration in structures/cm^2.

18.1.2 The analytical sensitivity.

18.1.3 Types of asbestos present.

18.1.4 Number of asbestos structures counted.

18.1.5 Effective filtration area.

18.1.6 Average size of the TEM grid openings that were counted.

18.1.7 Number of grid openings examined.

18.1.8 Sample dilution used.

18.1.9 Area of the surface sampled.

18.1.10 Listing of size data for each structure counted.

18.1.11 A copy of the TEM count sheet or a complete listing of the raw data. An example of a typical count sheet is shown in Appendix X1.

18.2 Determine the amount of asbestos in any accepted sample using the following formula:

$$\frac{EFA \times 100\ \text{mL} \times \#STR}{GO \times GOA \times V \times SPL} = \text{asbestos structures/cm}^2 \qquad (1)$$

where:

$\#STR$ = number of asbestos structures counted,
EFA = effective filter area of the final sampling filter, mm^2,
GO = number of grid openings counted,
GOA = average grid opening area, mm^2,
SPL = surface area sampled, cm^2, and
V = volume of sample filtered in step 10.4.9, representing the actual volume taken from the original 100 mL suspension, mL.

19. Quality Control/Quality Assurance

19.1 In general, the laboratory's quality control checks are used to verify that a system is performing according to specifications regarding accuracy and consistency. In an analytical laboratory, spiked or known quantitative samples are normally used. However, due to the difficulties in preparing known quantitative asbestos samples, routine quality control testing focuses on re-analysis of samples (duplicate recounts).

19.1.1 Re-analyze samples at a rate of 1/10 of the sample sets (one out of every ten samples analyzed not including laboratory blanks). The re-analysis shall consist of a second sample preparation obtained from the final filter.

19.2 In addition, quality assurance programs must follow the criteria shown in the *USEPA Asbestos-Containing Materials in Schools* document (4) and in the *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* document (6). These documents describe sample custody, sample preparation, blank checks for contamination, calibration, sample analysis, analyst qualifications, and technical facilities.

20. Calibrations

20.1 Perform calibrations of the instrumentation on a regular basis, and retain these records in the laboratory, in accordance with the laboratory's quality assurance program.

20.2 Record calibrations in a log book along with dates of calibration and the attached backup documentation.

20.3 A calibration list for the instrument is as follows:

20.3.1 *TEM:*

20.3.1.1 Check the alignment and the systems operation. Refer to the TEM manufacturer's operational manual for detailed instructions.

20.3.1.2 Calibrate the camera length of the TEM in electron diffraction (ED) operating mode before ED patterns of unknown samples are observed. Camera length can be measured by using a carbon coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thick gold films will tend to mask weak diffraction spots from the fibrous particles. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings from thick films are unnecessary. Alternatively, a gold standard specimen can be used to obtain an average camera constant calculated for that particular instrument and can then be used for ED patterns of unknowns taken during the corresponding period.

20.3.1.3 Perform magnification calibration at the fluorescent screen. This calibration must be performed at the magnification used for structure counting. Calibration is performed with a grating replica (7.47) (for example, one containing at least 2160 lines/mm).

(*a*) Define a field of view on the fluorescent screen. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

(*b*) Frequency of calibration will depend on the service history of the particular microscope.

(*c*) Check the calibration after any maintenance of the microscope that involves adjustment of the power supply to the lens or the high voltage system or the mechanical disassembly of the electron optical column (apart from filament exchange).

(*d*) The analyst must ensure that the grating replica is placed at the same distance from the objective lens as the specimen.

(*e*) For instruments that incorporate a eucentric tilting specimen stage, all specimens and the grating replica must be placed at the eucentric position.

20.3.1.4 The smallest spot size of the TEM must be checked.

(*a*) At the crossover point, photograph the spot size at a screen magnification of 15 000 to 20 000X. An exposure time of 1 s is usually adequate.

(*b*) The measured spot size must be less than or equal to 250 nm.

20.4 *EDXA:*

20.4.1 The resolution and calibration of the EDXA must be verified.

20.4.1.1 Collect a standard EDXA Cu peak from the Cu grid.

20.4.1.2 Compare the X-ray energy versus channel number for the Cu peak and be certain that readings are within ±10 eV.

20.4.2 Collect a standard EDXA of crocidolite asbestos (NIST SRM 1866).

20.4.2.1 The elemental analysis of the crocidolite must resolve the Na peak.

20.4.3 Collect a standard EDXA of chrysotile asbestos.

20.4.3.1 The elemental analysis of chrysotile must resolve both Si and Mg on a single chrysotile fiber.

20.5 Ultrasonic bath calibration shall be performed as follows:

20.5.1 Fill the bath water to a level equal to the height of suspension in the glass sample container that will be used for the dust analysis. Operate the bath until the water reaches the equilibrium temperature.

20.5.2 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.3 Place the sample container in the water in the ultrasonic bath (with the power turned off). After 60 s, remove the glass container and record its temperature.

20.5.4 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.5 Place the second sample container into the water in the ultrasonic bath (with the power turned on). After 60 s, remove the glass container and record its temperature.

20.5.6 Calculate the rate of energy deposition into the sample container using the following formula:

$$R = 4.185 \times \sigma \times \rho \times \frac{(\theta_2 - \theta_1)}{t} \quad (2)$$

where:

4.185 = Joules/cal,

R = energy deposition, watts/mL,

θ_1 = temperature rise with the ultrasonic bath not operating, °C,

θ_2 = temperature rise with the ultrasonic bath operating, °C,

t = time in seconds, 60 s (20.5.3 and 20.5.5),

σ = specific heat of the liquid in the glass sample container, 1.0 cal/g, and

ρ = density of the liquid in the glass sample container, 1.0 g/cm^3.

20.5.7 Adjust the operating conditions of the bath so that the rate of energy deposition is in the range of 0.08 to 0.12 MW/m^3, as defined by this procedure.

21. Precision and Bias

21.1 *Precision*—The precision of the procedure in this test method is being determined using round robin data from participating laboratories.

21.2 *Bias*—Since there is no accepted reference material suitable for determining the bias of the procedure in this test method, bias has not been determined (see Specification D 3670).

NOTE 3—Round robin data is under development and will be presented as a research report.

22. Keywords

22.1 asbestos; microvacuuming; settled dust; TEM

APPENDIX

(Nonmandatory Information)

X1. DUST SAMPLE ANALYSIS

X1.1 See Figs. X1.1 and X1.2 for the dust analysis worksheet and the TEM count sheet.

DUST SAMPLE ANALYSIS

Client: ______

Sample ID: ______

Job Number: ______

Date Sample Analyzed: ___ - ___ - ___

Number of Openings/Grids Counted: ___ | ___

Grid Accepted, 600X: Yes No

Percent Loading: ______ %

Grid Box #1: ______

Accelerating Voltage: ______

Indicated Mag: ______ KX

Screen Mag: ______ KX

Microscope: 1 2 3 4 5

Filter Type: ______

Filter Size: ______

Filter Pore Size (μm): ______

Grid Opening: 1) ______ μm x ______ μm

2) ______ μm x ______ μm

Analyst: ______

Reviewer: ______

Counting Rules: AHERA LEVEL II

Calculation Data:

Effective Filter Area in mm^2:	(EFA)	______
Number of Grid Openings Counted:	(GO)	______
Avderage Grid Opening Area in mm^2:	(GOA)	______
Volume of sample Filtered in ml:	(V)	______
Surface area Sampled in cm^2:	(SPL)	______
Number of Asbestos Structures Counted:*	(#STR)	______

* If the number of asbestos structures counted is less than or equal to 4, enter 4 structures as the limit of detection here.

FORMULA FOR CALCULATION OF ASBESTOS STRUCTURES "DUST" PER CM^2:

$$\frac{EFA X\ 100\ X\ \#STR}{GO\ X\ GOA\ X\ VX\ SPL} = \text{(Asbestos Structures per cm}^2\text{)}$$

Results for Total Asbestos Structures: ______
(Structures per cm^2)

Results for Structures ≥ microns: ______
(Structures per cm^2)

Job Number: ____________________

Structure #	Grid # Square #	Type	Structure	Length Microns	Width Microns	Confirmation		
						Morph.	SAED	EDS

Note: Keys to Abbreviations Used in Figure:

Type:

- C = Chrysotile
- AM = Amosite
- CR = Crocidolite
- AC = Actinolite
- TR = Tremolite
- AN = Anthophyllite
- N = Non Asbestos

Structure:

- F = Fiber
- B = Bundle
- C = Cluster
- M = Matrix

Others:

- NSD = No Structures Detected
- Morph = Morphology
- SAED = Selected Area Electron Diffraction
- EDS = Energy Dispersive X-Ray Spectroscopy
- ER = Inter-Row Spacing
- NP = No Pattern

FIG. X1.2 TEM Count Sheet

REFERENCES

(1) Steel, E. and Wylie, A., "Mineralogical Characteristics of Asbestos," in *Geology of Asbestos Deposits*, Riordon, P. H., Ed., SME-AIME, 1981, pp. 93–101.

(2) Zussman, J., "The Mineralogy of Asbestos," in *Asbestos: Properties, Applications and Hazards*, John Wiley and Sons, 1979, pp. 45–67.

(3) Leake, B. E., "Nomenclature of Amphiboles," *American Mineralogist*, Vol 63, 1978, pp. 1023–1052.

(4) "*USEPA Asbestos-Containing Materials in Schools: Final Rule and Notice*," *Federal Register*, 40 CFR Part 763, Appendix A to Sub-part E, Oct. 30, 1987.

(5) OSHA, *OSHA Technical Manual, OSHA Instruction CPL 2-20B*, Directorate of Technical Support, U.S. Department of Labor, Washington, DC 20210, Feb. 5, 1990, pp. 1–8 to 1–11.

(6) *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis*, NISTIR, August 1989, pp. 89–4137.

(7) Yamate, G., Agarwall, S. C. and Gibbons, R. D., "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy," EPA Draft Report, Contract No. 68-02-3266, 1984.

(8) Chatfield, E. J., and Dillon, M. J., "Analytical Method for the Determination of Asbestos in Water," EPA No. 600/4-83-043, 1983.

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Appendix D
Logbook Pages

6/26/03 cont.

1400
~~1345~~ pump 3143 with cassette #
T3 6/26/03 CS-12504 ~~placed on~~ TB 6/26/03 turned
on for 30 min Exc. sample.

1415 - pump 3143 with cassette # CS-12504
turned off and removed from T. Burgesser.
post calibration of pump 3143
is 2.1 L/min. Ave: 2.12 L/min for 30 min.

2000 · Turned off pump 3144 with cassette
CS-12501. Post calibration = 2.1 L/min.

2015 · Turned off pump 2702 with
cassette # CS-12503. Post calibration
= 2.05 l/min ave 2.07 L/min.

2030 - Exit lab

Todd B

6/27/03 Author Todd Burgesser

CSF ops plan 4/25/03 1551 L.bby orkeus6
Neg exposure sampling

0600 Calibrate pump 3143 with cassette
~~1256~~ T3 6/27/03 CS-12506 in line →
Flow rate = 2.1 L/min.
Calibrated pump 3142 with cassette
CS-12507 ~~CS-12508~~ in line flow rate =
2.12 L/min. →

0730 - Placed pump 3143 on T Clemans
with cassette CS-12505 ~~12506~~ TB 6/27/03 in line.
and started the pump.

0800 Placed pump 3142 on T Burgesser
with cassette CS-12507 ~~CS-12508~~ TB 6/27/03 in
line. and turned on pump

0830 D Pope and B McKee pull samples
for Big Pull of 500+ samples.

1000 - Enter CSC info Inventory 1072

Todd Burgesser

6/27/03 cont.

calibrate pump 2782 w label
cassette no. ~~2782~~ TB 2.1 L/min. CS-12506

1130 Received samples from Fed-X 2 coolers.

1135 - Exit for Lunch.

1137 Turn of pump

1207 Turn pum back on. 3142 -

Att TB 6/27/03

1305 - Attach pump 2572 to T Clemmons
for the 30-minute exp. with cassette

CS- 12506 TB 6/30/03

1335 - Removed cassette CS-12506 and
pump 2572 from T Clemmons.
Flow Rate 2.12 L/min.

1345 - Calibrate pump 2572 with casset
CS- 12508 TB 6/30/03 for T. Burgesser
2.05 L/min

30 minute exposure, ~~casset #~~ TB 6/27/03

Todd Burgesser
6/27/03

6/27/03 cont. -

1350 - Attach pump ~~CS-~~ TB 6/27/03 2572
to T. Burgesser and starte
with cassette CS-12508.

1410 - Remove pump ~~CS~~ TB 6/27/03 2572
with cassette CS-12508 from
T. Burgesser - Calibrature = ~~2.12 L/min~~ 2.0 6/27/03

1530 - Remove pump 3143 with the cassette
from T Clemmons.

CS- 12505 TB 6/30/03

Pump post calibration = 2.02 L/min
for average of 2.05 L/min.

1600 - Removed pump 3142 with cassette
CS-12507 **CS- 12507** TB 6/30/03

Post calibration of pump = 2.1 L/min
for average of 2.11 L/min.

Todd Burgesser
Todd Burgesser
6/27/03

104 6/27/03 cont.

Placed all cassettes in the baggy for use at the lab.

1500 - Placed all samples that were prepped previously in the bags ready for shipment.

1830 - Shipped out COCs 1078, 1079 and 1080 to Ellis Westmont for TLM/Grav. Analysis. COC 1079 is Dupes, COC 1080 is gravimetric.

1900 - Exit lab for Fed-x and home.

Todd

6/30/03 Author Todd Burgess 105

Company Plan: CSF Soil operation Plan 4/25/03 ISSI Lab 61 Rev 6. →

0600 - Arrived at lab. Data Entry of all samples ground over the last week. QC of FSDS and Data entry. QC check of Calibration Logs. →

0630 - Calibration of pump 3143 with cassette CS-12509

CS- 12509 TB 6/30/03 in line.

flow Rate = 2.05 l/min.

0635 - Calibration of pump 3146 with TB 6/30/03 a cassette CS-12510 in line

CS- 12510 TB 6/30/03 flow = 2.1 l/min.

0640 - Calibration of pump 3142 with cassette # CS-12511 in line.

CS- 12511 TB 6/30/03 flow = 2.11 L/min

Todd Burgess 6/30/03

 6/30/03 cont.

0640. Calibration of pump ~~CS-12512~~ TB 6/30/03 2752 with cassette CS-12512 in line. flow = 2.1 L/mm.

CS- 12512 TB 6/30/03

0645 - Opened up two cassetts, CS-12513 and CS-12514 for personal air blanks.

CS- 12513 TB 6/30/03 **CS- 12514** TB 6/30/03

0650 - Calibration of hi vol pump # 1088 with cassette # CS-12515-

CS- 12515 TB 6/30/03 Flow = 8.58 L/min. Pump in office

0655 - Calibration of pump 889-1055 with cassette # CS-12516 in line.

CS- 12516 TB 6/30/03 flow = 8.52 L/min. pump in Sample Receiving.

Todd Burgesse
Todd Burgesse
6/30/03

6/30/03 cont.

0700 Calibration of pump 0998 with cassette # CS-12517

CS- 12517 TB 6/30/03 flow = 8.56 L/min.

Pump placed in lab on the North Side. →

0705 - Calibration of pump # 0890 with cassette # CS-12518 in line,

CS- 12518 TB 6/30/03 flow = 8.58 L/min.

Pump placed in sample storage.

0710 - Opened up ~~pumps~~ TB 6/30/03 cassettes CS-12519 and CS-12520 for 30 seconds.

CS- 12519 TB 6/30/03 **CS- 12520** TB 6/30/03

0720 - All High vol pumps turned on.

These are our blank air blanks.

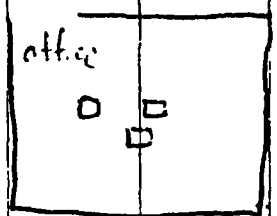
Todd Burgesse
Todd Burgesse
6/30/03

108 6/30/03 cont.

0715 ~~CS~~ TB ~~D~~ TB 6/30/03 Collection of Dust sample CS-12521 with pump # 3146. pump calibrated to 2.1 L/min.

CS- 12521 TB 6/30/03

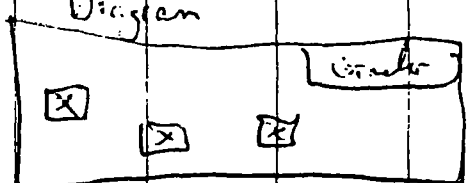
Diagram: 3 100 cm² areas for two minutes each.



0726 Collection of Dust samples in the hood # 2 in 3 areas with cassette # CS-12522 in line. calibration flow = 2.1 L/min.

CS- 12522 TB

Diagram



Todd Burgesser 6/30/03

6/30/03 cont.

0733 Collection of dust sample CS-12523 in the main lab by the hoods. 300 cm² for ~~60~~ 3 min.

CS- 12523 TB 6/30/03

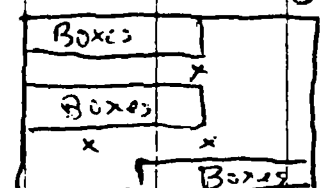
0736 Stop collection of CS-12523.

~~0717~~ 0720 - Pump 3143 with cassette CS-12509 placed on T. Clemens and turned on.

0720 - Pump 3142 - with cassette CS-12511 Attached to T. Burgesser and turned on.

0740 - collection of Dust sample CS-12524 on the floor in the Sample storage garage. diagram bellow

CS- 12524 TB 6/30/03



Collection of 300 cm² in three areas 100 cm² each.

Todd EB 6/30/03

6/30/03 cont

0730 - Turned on all high volume pumps for ambient air sampling.

0755 - Collection of dust samples off the top of 3 boxes (100 cm² each) in the sample storage room. CS-12525.

CS- 12525 TB 6/30/03 flow = 2.1 L/min.

0801 - Collection of dust from the 3 desks (100 cm²) each in the office. for 6 minutes total with cassette # CS-12526.

CS- 12526 TB 6/30/03 flow = 2.1 L/min

0810 - Collection of two Dust blanks cs #s

CS- 12529 6/30/03 TB **CS- 12528** TB 6/30/03

Opened cassette for 30 sec each.

0811 - Placed pump # 2752 with cassette 12512 on T. Burgesser for 30-minute exc.

Todd Burgesser 6/30/03

6/30/03 cont.

0831 - Turned off the pump for the 30-minute excursion sample. Pump ~~3146~~ 2752 TB 6/30/03 calibrated at 2.0 L/min for average with cassette CS-12512 of 2.05 L/min.

0835 - Attached pump CS-~~12~~510 with pump 3146 to T. Clemans for the 30 minute excursion sample and started the pump.

~~08~~ TB 6/30/03 0905 - Removed the pump (3146) with CS-12510 from T. Clemans. 30 min excursion sample. Pump calibration at 2.1 L/min.

Placed the sample in the Bag.

0900 - Exit to the office to get the FSDS to start filling out.

Transfer the pump to K Sloan for 30+ minute.

1041 - ~~Samples~~ TB Back at lab and transfer pump from K. Sloane to T Burgesser.

Todd Burgesser 6/30/03

2 6/30 Cont:

1045 - T Clemans turned of pump 3143 →

1047: Start filling out FSDS for the negative exposure assessments →

1107 - Transferred the SampleCorrelator pump to Andy Richardson to wear for the day. →

1108 Put on T. Clemans pump 3143 to wear for the granuly operations

1320 - Turned of all pumps for the sktoring air samples →

Post calibration of pumps with Cassette in line is as follows;

Pump 1088 w CS-12515 = 8.52

889-1055 w CS-12516 = 8.50

0998 w CS-125A = 8.52

0890 w CS-12518 = 8.52

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6/30/03 Cont.

1320 - Remove Pump 3143 from T Burgess with cass CS12509. Post cal. bration = 2.0 L/min.

Remove pump to 3142 with cassette CS-12511 w from A. Gordon. Flow = 2.0 L/min. →

1600 - Exit LAB for office to fill COCs. →

Todd Burgess

Todd Burgess

6/30/03